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- (54) PROCEDE DE PRODUCTION DE DETERGENT POUR LESSIVE ET PASTILLES DE PRODUIT DE NETTOYAGE
- (54) PROCESS FOR PRODUCING LAUNDRY DETERGENT AND CLEANING PRODUCT TABLETS

(57) A production process for laundry detergent and cleaning product tablets which is superior to the existing tabletting and extrusion technology in respect of the protection of the ingredients against thermal loads, pressure and shearing, which is less complex in terms of the apparatus and more favorable in terms of process economics, and which permits higher throughputs and can be used without great effort for the production of tablets with three or more phases as well, comprises producing (a) deformable mass(es), supplying this mass with a pressure less than 40 bar to emergence apertures, and cutting and hardening the emergent material strands.

Abstract

A production process for laundry detergent and cleaning product tablets which is superior to the existing tabletting and extrusion technology in respect of the protection of the ingredients against thermal loads, pressure and shearing, which is less complex in terms of the apparatus and more favorable in terms of process economics, and which permits higher throughputs and can be used without great effort for the production of tablets with three or more phases as well, comprises producing (a) deformable mass(es), supplying this mass with a pressure less than 40 bar to emergence apertures, and cutting and hardening the emergent material strands.

Process for producing laundry detergent and cleaning product tablets

Field of the Invention

The present invention relates to a new process for producing single-phase and multiphase laundry detergent and cleaning product tablets.

Background of the Invention

Laundry detergent and cleaning product tablets have 10 been widely described in the prior art and are enjoying increasing popularity among users owing to the ease of dosing. Tableted cleaning products have a number of advantages over their powder-form counterparts: They are easier to dose and to handle, and have storage and 15 transport advantages owing to their compact structure. Consequently, there exists an extremely broad prior art relating to laundry detergent and cleaning product tablets, which is also reflected in an extensive patent 20 literature. At an early stage, the developers of products in tablet form hit upon the idea of using tablet regions of different composition to release certain ingredients only under defined conditions in the course of washing or cleaning in order to improve the end result. Tablets which have become established 25 in this context are not only the core/sheath tablets and ring/core tablets, which are sufficiently well from pharmacy, but also, in particular, multilayer tablets, which are nowadays available for many areas of washing and cleaning or of hygiene. 30 Visual differentiation of the products is also becoming increasingly important, so that single-phase single-color tablets in the field of washing cleaning have been largely displaced by multiphase tablets. Common current market forms include two-layer 35 tablets having a white and a colored phase or having two differently colored layers. In addition, exist inlay tablets, ring-core tablets, laminated tablets, etc., whose importance at present is fairly minor.

The preparation of said tablets always comprises at least one tableting step, in which a particulate premix is converted under pressure into a compact tablet. In the case of the abovementioned two-layer tablets, sheath/core tablets, etc., different premixes are pressed onto and/or into one another. In addition, there exist proposals to produce tablets by means of conventional compression technology and to fill cavities in these tablets with melts or the like in order to obtain composition tablets comprising compressed and noncompressed fractions.

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A further process utilized to produce compact laundry detergent and cleaning product pieces is that of extrusion. Here, a premix is plasticated under high pressures and discharged through perforated molds, followed by shaping by means of cutting, and by optional aftertreatment. In contrast to tableting, where the particle bed is, so to speak, "sintered", and where the tablets still possess a hollow volume, the high pressures of 100 bar or more during extrusion lead to very compact particles or pieces whose internal hollow volume is greatly reduced.

Multilayer cleaning product tablets for machine dishwashing are described, for example, in European Patent Application EP 224 128 (Henkel KGaA). The two layers have solubility differences, which leads to advantageous performance properties.

Multiphase cleaning tablets for the WC are described,
for example, in EP 055 100 (Jeyes Group). This document
discloses toilet cleaning blocks comprising a shaped
body, consisting of a slow-dissolving cleaning product
composition, into which a bleach tablet has been

embedded. At the same time, this document discloses the very wide variety of design forms of multiphase tablets. In accordance with the teaching of this document, the tablets are produced either by inserting a compressed bleach tablet into a mold and casting the cleaning product composition around this tablet, or by casting part of the cleaning product composition into the mold, followed by the insertion of the compressed bleach tablet and, possibly, subsequent overcasting with further cleaning product composition.

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In addition, EP 481 547 (Unilever) describes multiphase cleaning product tablets which are intended for use for machine dishwashing. These tablets have the form of core/sheath tablets and are produced by stepwise compression of the constituents: first of all, a bleach composition is compressed to form a tablet, which is placed in a die which is half-filled with a polymer composition, this die then being filled up with further polymer composition and compressed to form a bleach tablet provided with a polymer sheath. The process is

subsequently repeated with an alkaline cleaning product

composition, so as to give a three-phase tablet..

25 Another route to producing visually differentiated laundry detergent and cleaning product tablets described in International Patent Applications W099/-06522, W099/27063 and W099/27067 (Procter & Gamble). According to the teaching of these documents, a tablet is prepared which has a cavity that is filled with a 30 solidifying melt. Alternatively, a powder is introduced and is fixed in the cavity by means of a coating layer. A common feature of all three applications is that the region filling out the cavity should not be compressed, 35 the intention is in this way to protect "pressure-sensitive" ingredients.

Both tableting and extrusion lead to a high pressure load on the premixes for processing, which makes it difficult or impossible to incorporate pressure-sensitive ingredients. Also, the production of tablets with three or more phases is no longer possible without problems using either process, since the technical expense increases greatly as the number of phases goes up.

The extrusion or coextrusion of two or more premixes is virtually impossible when there are large differences in the proportion of the individual phases. The conventional tableting of multilayer tablets likewise reaches its limits in the field of laundry detergent and cleaning product tablets if one layer is intended to comprise only a small fraction of the total tablet. Below a certain layer thickness, compression of a layer adhering to the remainder of the tablet becomes increasingly difficult.

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Summary of the Invention

It is an object of the present invention, then, to provide a production process for single-phase and multiphase tablets in which even pressure-sensitive ingredients may be accommodated in delimited regions, without any restrictions on the size of the delimited region in relation to the total tablet. At the same time, moreover, firstly there ought to be visual differentiation from conventional two-layer tablets and secondly the production of the tablets ought to function reliably without great technical effort even in mass production without the tablets suffering from stability drawbacks and without the fear of dosing inaccuracies.

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In particular, it is an object of the present invention to provide a new production process for laundry detergent and cleaning product tablets which is superior to the existing tableting and extrusion technology in respect of the protection of the ingredients against thermal loads, pressure and shearing, which is less complex in terms of the apparatus and more favorable in terms of process economics, and which permits higher throughputs. In addition, the process should be able to be used without great effort for the production of tablets with three or more phases as well.

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It has now been found that the low-pressure strand processing of deformable, curable masses is suitable for producing laundry detergent and cleaning product tablets, and does so while meeting the abovementioned profiles of requirements.

The invention provides a process for producing laundry detergent and cleaning product tablets, which involves preparing (a) deformable mass(es), supplying said mass(es) with a pressure below 40 bar to emergence apertures, cutting the emerging material strands to tablet dimensions, and hardening them.

Preferably, the deformable masses which harden after deformation are supplied to the emergence apertures at even lower pressures, in order to protect pressuresensitive ingredients. Preferred processes are those wherein the deformable mass(es) is (are) supplied to the emergence apertures with a pressure below 35 bar, preferably below 30 bar, with particular preference below 20 bar, and in particular below 10 bar.

Depending on the final processing of the deformable masses (see below) and on the configuration of the processing machines, it is also possible to realize even lower pressures, or a procedure without pressure is possible. Processes wherein the deformable mass(es) is (are) supplied to the emergence apertures with a

pressure below 8.5 bar, preferably below 7.5 bar, with particular preference below 6.5 bar, and in particular below 5 bar, are a further important embodiment of the present invention.

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Detailed Description of the Invention

Below, technical apparatus parameters and technical features of the process of the invention will be described, before the ingredients and physical parameters of the masses for processing are dealt with.

provides The the invention process of for the processing of deformable masses which harden orsolidify after shaping to give compact tablets. contrast to the extrusion of laundry detergents and cleaning products, where solid, free-flowing premixes are plasticated and shaped by high pressures, process of the invention is operated at low pressures and starts from deformable masses. These deformable masses are not particulate, but are paste-like or plastic and harden after shaping.

A procedure preferred in the context of the present invention for supplying the deformable masses to the emergence apertures comprises drawing them in between two rolls which have opposite directions of rotation. By this means, the mass between the rolls is conveyed under low pressure, depending on the width of the nip between the rolls and on the roll speed, in the direction of the emergence apertures. Depending on the number of roll pairs and emergence apertures, and on the design of these apertures, this results in single-phase or multiphase material strands which may have different shapes and/or colors. These material strands are cut into sections of predetermined length, and the individual strand sections are hardened to give the finished laundry detergent and cleaning product tablet.

Single-phase tablets are advantageously produced by supplying a deformable mass with one roll pair to an emergence aperture. Preferred processes are those wherein a deformable mass is drawn in between two rolls, discharged as a material strand from emergence apertures, cut to the desired tablet dimension, and hardened.

Apparatus suitable for the preferred process is obtain-10 able, for example, from the company Hosokawa Bepex GmbH under the name "Drehstab-Walzenpresse DP" [rotary-rod roll press DP]. The emergence apertures of apparatus may, for example, be circular, triangular, square, rectangular, heart-shaped, crescent-shaped, 15 etc., in design. The first-mentioned apertures then give rise to cylindrical, prismatic, cuboid, tetragonal or orthorhombic tablets. The drawings, in Figures 27 and also 29 to 42, show by way of example some possible designs for emergence apertures.

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It is likewise possible to rotate the material strands following emergence from the apertures in the shapable state before they are cut to the desired tablet dimensions. This produces tablets having irregular, spiral side faces, which offer particular visual attractiveness.

Two-phase tablets may be produced correspondingly using two roll pairs. Preferred processes for this purpose are those wherein two deformable masses of different composition are drawn in between two roll pairs and discharged as filled, hollow or multi-ply material strands from emergence apertures, cut to the desired tablet dimension, and hardened. It is of course also possible to process two masses of identical composition in a similar way. In this case this is done not for separation of active substances or to achieve specific washing and cleaning effects, but instead for visual

attractiveness. Apparatus suitable for such processes of the invention is again available from the company Hosokawa Bepex GmbH under the name "Doppel-Drehstab-Walzenpresse DDP" [double rotary-rod roll press DDP]. The emergence apertures of such apparatus may arranged alongside or inside one another, producing multi-ply or multiphase tablets. Figures 15 to 26 and 28 show by way of example some cross sections of emergence apertures for different masses. In Figures 10 15, 16, 17, 19, 21, 23, and 25, there result strands and tablets in which one part, apart from the cut faces, is completely enclosed by the other part. The other figures mentioned show strands or respectively, in which one part is embedded on or only 15 partly in the other part. Here again, rotation of the material strands prior to cutting to length is possible in order to achieve particular visual effects.

The process of the invention may also be utilized without problems to produce three-phase tablets. Completely
in analogy to the remarks made so far, such processes
of the invention are implemented by drawing in three
plastically deformable masses of different composition
between three roll pairs and discharging them as
singly, doubly or triply filled, hollow, two- or threeply material strands from emergence apertures, cutting
them to the desired tablet dimension, and hardening
them.

Here again, it is of course possible to process two or even three masses of identical composition in a similar manner. This is then used in turn not (only) to separate active substances or to achieve particular washing and cleaning effects, but instead for visual attractiveness. Apparatus suitable for the production of three-phase tablets, as well, is available from the company Hosokawa Bepex GmbH under the name "Dreifach-Drehstab-Walzenpresse DP/3" [triple rotary-rod roll

press DP/3]. The emergence apertures of such apparatus may be arranged alongside or inside one another, producing multi-ply or multiphase tablets. Figures 1 to 14 show by way of example some cross sections of emergence apertures for different masses. Here again, in turn, the material strands may be rotated prior to cutting to length in order to obtain particular visual effects.

10 The possibilities for discharging a plurality of material strands onto, alongside, over, under or into one another from the apparatus are unlimited, so that even tablets with four or more phases may be produced in a simple manner. Since the apparatus and the associated die systems are of simple and robust construction, a changeover of product shape, and adaptation to differmarket requirements, are possible rapidly and without complication. Changes in shape on the resultant tablets may also be brought about without problems by 20 means of the appropriate treatment of the material strands prior to cutting. If, for example, in accordwith Figure 1, three material strands discharged onto one another from three emergence apertures having a circular cross section, then, after cutting to length, tablets are produced which have the 25 form of three stacked cylinders. By simple rotation of the three material strands about their longitudinal axis prior to cutting to length, laundry detergent and cleaning product tablets are obtained which have the 30 form of segments turned in toward one another suggest rigging or braids. The flexibility of process of the invention in terms of the changeover of shapes and esthetic design is therefore far greater than that of the processes known to date.

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In the production of multiphase tablets, the ratio of these phases to one another may be chosen freely; from esthetic standpoints, it may be advantageous if one phase accounts for at least 1/100, preferably at least 1/20 and in particular at least 1/10 of the volume or the weight of the other phase(s). In preferred process end products, the weight ratio of the masses to one another is in the range from 1:1 to 1:100, preferably from 1:2 to 1:75, and in particular from 1:2.5 to 1:30 (two-phase tablets) or, respectively, in the range from 1:1:1 to 1:100:100, preferably from 1:1:2 to 1:75:75, and in particular from 1:1:2.5 to 1:30:30 (three-phase tablets). The ratio of the surface areas of the individual tablet phases is preferably within similar ranges.

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Depending on the final processing of the deformable masses (see below), i.e., depending on the ingredients 15 and the physical parameters of the masses for processing, it is possible to achieve throughputs of differing levels, which further depend on the size of emergence apertures. It is preferred in this context to keep within certain emergence rates for the material 20 strands. In preferred processes the material strands are discharged from the emergence apertures at a rate of from 0.2 m/min to 30 m/min, preferably from 0.25 m/min to 20 m/min, with particular preference from 25 0.5 m/min to 15 m/min, and in particular from 1 m/min to 10 m/min.

In principle, the process of the invention is not limited as regards the shape and size of the emergence apertures. In view of the products to be manufactured and their size and mass, which in the case of such products is usually in the range from 5 to 500 g, preferably from 10 to 250 g, with particular preference from 15 to 100 g, and in particular between 20 and 50 g, preference is given to processes wherein the emergence apertures have aperture areas of from 50 mm² to 2500 mm², preferably from 100 mm² to 2000 mm², with particular preference from 200 mm² to 1500 mm², and in

particular from 300 mm² to 1000 mm², with especial preference from 350 mm² to 750 mm².

However, it is possible to drop below these values for individual emergence apertures, for example, if the purpose of one emergence aperture is to place a thin "tube" over another strand, thereby, so coating the latter. Strand cross sections of this kind are sketched, for example, in Figures 15, 17, 21 and 23, it being entirely possible in each case for the external part to be thinner. In that case, the finished tablet contains strands which apart from the end faces (cut faces) are coated, from which effects may be achieved in terms of delayed or accelerated release. With the exception of such coated strands, however, preference is given to processes wherein the thickness of at least one of the material strands emerging from the emergence apertures is at least 5 mm, preferably at least 7.5 mm, and in particular at least 10 mm.

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The cutting to length of the material strands emerging from the emergence apertures may take place in accordance with the known processes of the prior art, for example, by means of rotating blades, lowerable cutters or wires, etc. The mass of the finished tablets is guided on the one hand by the size of the emergence apertures and on the other hand by the length of the cuts. Where conventional laundry detergent and cleaning product tablets are to be provided for customary utilities such as, for example, laundry detergent tablets or machine dishwashing detergent preference is given to processes wherein the material strands emerging from the emergence apertures are cut to a length of from 10 to 100 mm, preferably from 12.5 to 75 mm, with particular preference from 15 to 60 mm, and in particular from 20 to 50 mm.

Depending on the composition or on the desired end use, however, it is also possible to go above or below the abovementioned limits. Thus it is possible, for example, to process masses which are relatively poorly soluble after hardening and to cut them to lengths of from 100 to 1000 mm, preferably from 120 to 750 mm, and in particular from 150 to 500 mm. The hardened "rods" obtained in this way may then be accommodated in washing machines or dishwashers as depot blocks, where in each washing cycle a defined portion of the block is dissolved while the remainder remains in the machine or its dosing system for the next cleaning cycle.

After cutting to the desired tablet dimensions, 15 strand sections are hardened. Hardening is carried out differently (see below) depending on the composition of so that hardening may if the masses, desired be accelerated assisted by means of appropriate measures. It is possible, for example, to initiate or 20 accelerate reactive hardening by spraying activators onto the surface. In the case of radiation-hardening masses, exposure to radioactive rays may also utilized, as can UV radiation for UV-active masses. In preferred processes, hardening takes place by means of 25 internal and external drying and/or cooling, so that preferred processes are those wherein the hardening of the material strands, cut to tablet dimensions, assisted by superficial drying and/or cooling, in particular by blowing with cold air.

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Following the description of the preferred embodiments in terms of apparatus, there now follows a description of the deformable and hardening masses for processing. In this context, there is both a treatment of the composition and physical parameters and a description of possible hardening mechanisms.

The hardening of the deformable mass(es) may take place by different mechanisms, among which mention may be made of time-delayed water binding, cooling below the melting point, solvent evaporation, crystallization, chemical reaction(s), especially polymerization, and changes in the rheological properties as a result, for example, of altered shearing of the mass(es), as the most important hardening mechanisms, in addition to the already mentioned radiation hardening by means of UV, alpha, beta or gamma rays.

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In all cases, a deformable, preferably plastic, mass is prepared which may be shaped without great pressures. After shaping, hardening then takes place by means of suitable initiation or elapse of a certain period of time. If the masses processed have self-hardening properties without further initiation, then this must be taken into account in the course of processing in order to avoid instances of hardening during shaping and, consequently, blockages and disruptions to the process sequences.

In processes which are preferred in the context of the present invention, hardening of the deformable mass(es) takes place by means of time-delayed water binding.

Time-delayed water binding in the masses processed in accordance with the invention may in turn be realized in different ways. Appropriate in this context, for example, are masses which comprise hydratable, anhydrous raw materials, or raw materials in low states of hydration, which are able to undergo transition to stable, higher hydrates, and which further comprise water. The formation of the hydrates, which does not take place spontaneously, then leads to the binding of free water, which in turn leads to hardening of the masses. Subsequently, low-pressure shaping is no longer possible, and tablets stable to handling are produced

which may, if desired, be treated further and/or packaged.

Time-offset water binding may also take place, for example, by incorporating salts containing water of hydration, which when the temperature is increased dissolve in their own water of crystallization, into the masses. If there is a subsequent fall in temperature, the water of crystallization is bound again, leading to a loss of shapeability by simple means and to a solidification of the masses.

The swelling of natural or synthetic polymers is another time-delayed water binding mechanism which may be utilized in the context of the process of the invention. In this case, mixtures of unswellen polymer and suitable swelling agent, e.g., water, diols, glycerol, etc., may be incorporated into the masses, with swelling and hardening taking place after shaping.

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The most important mechanism of hardening by time-delayed water binding is the use of a combination of water with raw materials that are anhydrous or of low water content, which slowly hydrate. Particularly appropriate for this purpose are substances which contribute to the cleaning performance in the washing or cleaning process. In the context of the process of the invention, preferred ingredients of the deformable masses in this context are, for example, phosphates, carbonates, silicates, and zeolites.

It is particularly preferred if the resultant hydrate forms have low melting points, since in this way a combination of the hardening mechanisms by internal drying and cooling is achieved. Preferred processes are those wherein the deformable mass(es) comprise(s) from 10 to 95% by weight, preferably from 15 to 90% by weight, with particular preference from 20 to 85% by

weight, and in particular from 25 to 80% by weight, of anhydrous substances which pass by hydration into a hydrate form having a melting point below 120°C, preferably below 100°C, and in particular below 80°C.

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The deformable properties of the masses may be influenced by adding plasticizers such as polyethylene glycols, polypropylene glycols, waxes, paraffins, non-ionic surfactants, and so on. Further details of the classes of substance mentioned are given later on.

Raw materials for preferred incorporation into the deformable masses originate from the group of the phosphates, alkali metal phosphates being particularly preferred. For the preparation of the masses, these substances are used in anhydrous or low-water-content form, and the desired plastic properties of the masses are established using water and also optional plasticizers. After shaping, shaped and cut-to-length strands are then hardened by hydration of the phosphates.

Alkali metal phosphates is the collective term for the alkali metal (especially sodium and potassium) salts of the various phosphoric acids, among which metaphosphoric acids (HPO $_3$) $_n$ and orthophosphoric acid H $_3$ PO $_4$, in addition to higher-molecular-mass representatives, may be distinguished. The phosphates combine a number of advantages: they act as alkali carriers, prevent limescale deposits on machine components, and lime incrustations on fabrics, and additionally contribute to cleaning performance.

Sodium dihydrogen phosphate, NaH₂PO₄, exists as the dihydrate (density 1.91 g cm⁻³, melting point 60°) and as the monohydrate (density 2.04 g cm⁻³). Both salts are white powders of very ready solubility in water which lose the water of crystallization on heating and undergo transition at 200°C to the weakly acidic

diphosphate (disodium dihydrogen diphosphate, Na₂H₂P₂O₇) and at the higher temperature into sodium trimetaphosphate (Na₃P₃O₉) and Maddrell's salt (see below). NaH₂PO₄ reacts acidically; it is formed if phosphoric acid is adjusted to a pH of 4.5 using sodium hydroxide and the slurry is sprayed. dihydrogen phosphate (primary or monobasic potassium phosphate, potassium biphosphate, PDP), KH₂PO₄, white salt with a density of 2.33 g cm⁻³, has a melting of 253° [decomposition with formation potassium polyphosphate $(KPO_3)_x$, and is readily soluble in water.

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hydrogen phosphate Disodium (secondary sodium phosphate), Na₂HPO₄, is a colorless, crystalline salt 15 which is very readily soluble in water. It exists in anhydrous form and with 2 mol (density 2.066 g cm⁻³, water loss at 95°), 7 mol (density 1.68 g cm⁻³, melting point 48° with loss of 5 H_2O), and 12 mol of water (density 1.52 g cm⁻³, melting point 35° with loss of 20 5 H₂O), becomes anhydrous at 100°, and if heated more severely undergoes transition to the diphosphate Na₄P₂O₇. Disodium hydrogen phosphate is prepared by neutralizing phosphoric acid with sodium carbonate using phenolphthalein 25 solution as indicator. Dipotassium hydrogen phosphate (secondary or dibasic potassium phosphate), K₂HPO₄, is an amorphous white salt which is readily soluble in water.

Trisodium phosphate, tertiary sodium phosphate, Na₃PO₄, exists as colorless crystals which as the dodecahydrate have a density of 1.62 g cm⁻³ and a melting point of 73-76°C (decomposition), as the decahydrate (corresponding to 19-20% P₂O₅) have a melting point of 100°C, and in anhydrous form (corresponding to 39-40% P₂O₅) have a density of 2.536 g cm⁻³. Trisodium phosphate is readily soluble in water, with an alkaline reaction, and is prepared by evaporative concentration of a

solution of precisely 1 mol of disodium phosphate and 1 mol of NaOH. Tripotassium phosphate (tertiary or tribasic potassium phosphate), K_3PO_4 , is a white, deliquescent, granular powder of density 2.56 g cm⁻³, has a melting point of 1340°, and is readily soluble in water with an alkaline reaction. It is produced, for example, when Thomas slag is heated with charcoal and potassium sulfate. Despite the relatively high price, the more readily soluble and therefore highly active potassium phosphates are frequently preferred in the cleaning products industry over the corresponding sodium compounds.

Tetrasodium diphosphate (sodium pyrophosphate), Na₄P₂O₇, exists in anhydrous form (density 2.534 g cm⁻³, melting point 988°, 880° also reported) and as the decahydrate (density 1.815-1.836 g cm⁻³, melting point 94° with loss of water). Both substances are colorless crystals which dissolve in water with an alkaline reaction. Na₄P₂O₇ is formed when disodium phosphate is heated at > 200° or by reacting phosphoric acid with sodium carbonate in stoichiometric ratio and dewatering the solution by spraying. The decahydrate complexes heavy metal salts and water hardeners and therefore reduces the hardness of the water. Potassium diphosphate (potassium pyrophosphate), K₄P₂O₇, exists in the form of the trihydrate and is a colorless, hygroscopic powder of density 2.33 g cm⁻³ which is soluble in water, the pH of the 1% strength solution at 25° being 10.4.

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Condensation of NaH_2PO_4 or of KH_2PO_4 gives rise to higher-molecular-mass sodium and potassium phosphates, among which it is possible to distinguish cyclic representatives, the sodium and potassium metaphosphates, and catenated types, the sodium and potassium polyphosphates. For the latter in particular a large number of names are in use: fused or calcined phosphates, Graham's salt, Kurrol's and Maddrell's

salt. All higher sodium and potassium phosphates are referred to collectively as condensed phosphates.

The industrially important pentasodium triphosphate, Na₅P₃O₁₀ (sodium tripolyphosphate), is a nonhygroscopic, which white, water-soluble salt is anhydrous crystallizes with 6 H₂O and has the general formula $NaO-[P(O)(ONa)-O]_n-Na$ where n = 3. About 17 g of the anhydrous salt dissolve in 100 q of water at room temperature, at 60° about 20 g, at 100° around 32 g; after heating the solution at 100°C for two hours, orthophosphate and 15% diphosphate about 88 produced by hydrolysis. For the preparation of pentasodium triphosphate, phosphoric acid is reacted with sodium carbonate solution or sodium hydroxide solution in stoichiometric ratio and the solution is dewatered by spraying. In a similar way to Graham's salt and sodium diphosphate, pentasodium triphosphate dissolves insoluble metal compounds (including lime numerous Pentapotassium triphosphate, etc). K5P3O10 (potassium tripolyphosphate), is commercialized, example, in the form of a 50% strength by weight solution (> 23% P_2O_5 , 25% K_2O). The potassium polyphosphates find broad application in the laundry detergents and cleaning products industry. There also exist sodium potassium tripolyphosphates, which may likewise be used for the purposes of the present invention. These are formed, for example, when sodium trimetaphosphate is hydrolyzed with KOH:

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$$(NaPO_3)_3 + 2 KOH \rightarrow Na_3K_2P_3O_{10} + H_2O$$

These phosphates can be used in accordance with the invention in precisely the same way as tripolyphospate, potassium tripolyphosphate, or sodium mixtures of these mixtures of two; tripolyphosphate and sodium potassium tripolyphosphate, or mixtures of potassium tripolyphosphate and sodium potassium tripolyphosphate, or mixtures of sodium tripolyphosphate and potassium tripolyphosphate and sodium potassium tripolyphospate, may also be used in accordance with the invention.

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In preferred processes the deformable mass(es) comprise(s) phosphate(s), preferably alkali metal phosphate(s), with particular preference pentasodium or pentapotassium triphosphate (sodium or potassium tripolyphosphate), in amounts of from 20 to 80% by weight, preferably from 25 to 75% by weight, and in particular from 30 to 70% by weight, based in each case on the mass.

- Where phosphates are used as sole hydratable substances in the masses, the amount of added water should not exceed the water binding capacity thereof, in order to keep the free water content of the tablets low. Overall, processes which have been found to be preferred for observing the abovementioned limits are those wherein the weight ratio of phosphate(s) to water in the deformable mass is less than 1:0.3, preferably less than 1:0.25, and in particular less than 1:0.2.
- Further ingredients, which may be present instead of or in addition to phosphates in the deformable masses, are carbonates and/or hydrogen carbonates, preference being given to the alkali metal salts and, of these, particular preference to the potassium salts and/or sodium salts. In preferred processes, the deformable mass(es) comprise(s) carbonate(s) and/or hydrogen carbonate(s), preferably alkali metal carbonate(s), with particular preference sodium carbonate, in amounts of from 5 to 50% by weight, preferably from 7.5 to 40% by weight, and in particular from 10 to 30% by weight, based in each case on the mass.

Here again, the comments made above regarding the water content of the masses are applicable. Processes which have been found to be preferred, in particular, are those wherein the weight ratio of carbonate(s) and/or hydrogen carbonate(s) to water in the deformable mass is less than 1:0.2, preferably less than 1:0.15, and in particular less than 1:0.1.

Further ingredients which may be present instead of or in addition to the abovementioned phosphates and/or carbonates and/or hydrogen carbonates in the deformable masses are silicates, preference being given to the alkali metal silicates and, of these, particular preference to the amorphous and/or crystalline sodium and/or potassium disilicates.

Suitable crystalline, layered sodium silicates possess the general formula NaMSi_xO_{2x+1}·yH₂O, where M is sodium or hydrogen, x is a number from 1.9 to 4, y is a number from 0 to 20, and preferred values for x are 2, 3 or 4. Crystalline phyllosilicates of this kind are described, for example, in European Patent Application EP-A-0 164 Preferred crystalline phyllosilicates of formula indicated are those in which M is sodium and \boldsymbol{x} adopts the value 2 or 3. In particular, both β - and δ -sodium disilicates $Na_2Si_2O_5\cdot yH_2O$ are preferred, β -sodium disilicate, for example, being obtainable by described the process in International Application WO-A-91/08171.

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It is also possible to use amorphous sodium silicates having an Na₂O:SiO₂ modulus of from 1:2 to 1:3.3, preferably from 1:2 to 1:2.8, and in particular from 1:2 to 1:2.6, which are dissolution-retarded and have secondary washing properties. The retardation of dissolution relative to conventional amorphous sodium silicates may have been brought about in a variety of ways - for example, by surface treatment, compounding,

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compacting, or overdrying. In the context of this invention, the term "amorphous" also embraces "X-rayamorphous". This means that in X-ray diffraction experiments the silicates do not yield the sharp X-ray reflections typical of crystalline substances instead yield at best one or more maxima of scattered X-radiation, having a width of several degree units of the diffraction angle. However, good builder properties may result, even particularly good builder properties, if the silicate particles in electron diffraction experiments yield vague or even diffraction maxima. The interpretation of this is that the products have microcrystalline regions with a size of from 10 to several hundred nm, values up to max. 50 nm and in particular up to max. 20 nm being preferred. So-called X-ray-amorphous silicates of this which likewise possess retarded dissolution relative to the conventional waterglasses, described, for example, in German Patent Application DE-A-44 00 024. Particular preference is given to compacted amorphous silicates, compounded amorphous silicates, and overdried X-ray-amorphous silicates.

In processes which are preferred in the context of the present invention the deformable mass(es) comprise(s) silicate(s), preferably alkali metal silicates, with particular preference crystalline or amorphous alkali metal disilicates, in amounts of from 10 to 60% by weight, preferably from 15 to 50% by weight, and in particular from 20 to 40% by weight, based in each case on the mass.

Here again, the comments made above regarding the water content of the masses are applicable. Processes which have been found to be preferred are, in particular, those wherein the weight ratio of silicate(s) to water in the deformable mass is less than 1:0.25, preferably less than 1:0.2, and in particular less than 1:0.15.

Likewise suitable as an important component in the masses for processing in accordance with the invention are substances from the group of the zeolites. These substances constitute preferred builders especially in connection with the production of laundry detergent tablets. Zeolites have the general formula

$$M_{2/n}O \cdot Al_2O_3 \cdot x SiO_2 \cdot y H_2O$$

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in which M is a cation of valence n, x is greater than or equal to 2, and y may adopt values between 0 and 20. The zeolite structures are formed by linking of AlO₄ tetrahedra with SiO₄ tetrahedra, this network being occupied by cations and water molecules. The cations in these structures are relatively mobile and may be replaced to different degrees by other cations. The intercrystalline "zeolitic" water may be released, continuously and reversibly depending on zeolite type, while with certain types of zeolite structural changes are also associated with the release and/or uptake of water.

Within the structural subunits, the "primary binding units" (AlO₄ tetrahedra and SiO₄ tetrahedra) form socalled "secondary binding units", which possess the form of single or multiple rings. For example, various zeolites there are 4-, 6- and 8-membered rings (referred to as S4R, S6R and S8R), while other types are joined by way of four- and six-membered double-ring prisms (commonest types: D4R as a tetragonal and D6R as a hexagonal prism). These "secondary subunits" join different polyhedra, which are referred to using Greek letters. The most widespread in this context is a polyhedron composed of six squares and eight equilateral hexagons, which is referred to as " β ". Using these building units, it is possible to produce different zeolites. Known to date are 34 natural

zeolite minerals and approximately 100 synthetic zeolites.

The best-known zeolite, zeolite 4 A, is a cubic assembly of β cages linked by D4R subunits. It belongs to the zeolite structural group 3 and its three-dimensional network has pores of 2.2 Å and 4.2 Å in size; the formula unit in the unit cell may be described by Na₁₂[(AlO₂)₁₂(SiO₂)₁₂]·27 H₂O.

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In the process of the invention it is preferred to use zeolites of the faujasite type. Together with the zeolites X and Y, the mineral faujasite belongs to the faujasite types within the zeolite structural group 4, which is characterized by the double-hexagon subunit D6R (compare Donald W. Breck: "Zeolite Molecular Sieves", John Wiley & Sons, New York, London, Sydney, Toronto, 1974, page 92). In addition to the abovementioned faujasite types, the zeolite structural group 4 also includes the minerals chabazite and gmelinite and also the synthetic zeolite R (chabazite type), S (gmelinite type), L, and ZK-5. The two last-mentioned synthetic zeolites have no mineral analogs.

25 Zeolites of the faujasite type are composed of β cages linked tetrahedrally by way of D6R subunits, the β cages being arranged in a manner similar to the carbon atoms in diamond. The three-dimensional network of the faujasite-type zeolites used in the process of the 30 invention has pores of 2.2 and 7.4 Å; the unit cell includes, moreover, 8 cavities having a diameter of approximately 13 Å and may be described by the formula $Na_{86}[(AlO_2)_{86}(SiO_2)_{106}] \cdot 264$ H₂O. The network of zeolite X includes a cavity volume of approximately 50%, based on 35 the dehydrated crystal, which constitutes the largest empty space of all known zeolites (zeolite Y: approximately 48% cavity volume, faujasite: approximately 47% cavity volume). (All data from: Donald W. Breck:

"Zeolite Molecular Sieves", John Wiley & Sons, New York, London, Sydney, Toronto, 1974, pages 145, 176, 177.)

In the context of the present invention, the term "faujasite-type zeolite" denotes all three zeolites which form the faujasite subgroup of the zeolite structural group 4. In addition to zeolite X, therefore, zeolite Y and faujasite, and mixtures of these compounds, may be used in accordance with the invention, preference being given to straight zeolite X.

Mixtures or cocrystallizates of zeolites of 15 faujasite type with other zeolites, which need not necessarily belong to the zeolite structural group 4, may also be used in accordance with the invention, the advantages of the process of the invention being manifested particularly if at least 50% by weight of the 20 powdering agent comprises a faujasite-type zeolite. It is also conceivable, for example, to use the minimum amount of a faujasite-type zeolite (0.5% by weight, based on the weight of the tablet being produced) and to use conventional zeolite A as the remaining powder-25 ing agent. In any case, however, it is preferred for the powdering agent to consist exclusively of one or more faujasite-type zeolites, with zeolite X again being preferred.

- The aluminum silicates which are used with preference in the process of the invention are commercially available, and the methods of their preparation are described in standard monographs.
- 35 Examples of commercially available zeolites of the X type may be described by the following formulae:

 $Na_{86}[(AlO_2)_{86}(SiO_2)_{106}] \cdot x H_2O$,

 $K_{86}[(AlO_2)_{86}(SiO_2)_{106}] \cdot x H_2O_1$

 $Ca_{40}Na_{6}[(AlO_{2})_{86}(SiO_{2})_{106}] \cdot x H_{2}O_{7}$

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 $Sr_{21}Ba_{22}[(AlO_2)_{86}(SiO_2)_{106}] \cdot x H_2O_4$

in which x may adopt values of between 0 and 276, and which have pore sizes of from 8.0 to 8.4 Å.

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A product available commercially and able to be used with preference in the context of the present invention, for example, is a cocrystallizate of zeolite X and zeolite A (approximately 80% by weight zeolite X), which is sold by CONDEA Augusta S.p.A. under the brand name VEGOBOND AX® and may be described by the formula

 $nNa_2O \cdot (1-n) K_2O \cdot Al_2O_3 \cdot (2-2.5) SiO_2 \cdot (3.5-5.5) H_2O$.

Zeolites of the Y type are also commercially available and may be described, for example, by the formulae

 $Na_{56}[(AlO_2)_{56}(SiO_2)_{136}] \cdot x H_2O_1$

25 $K_{56}[(AlO_2)_{56}(SiO_2)_{136}] \cdot x H_2O$,

in which x stands for numbers between 0 and 276, and which have pore sizes of $8.0\ \text{\AA}.$

Preferred processes are those wherein the deformable mass(es) comprise(s) zeolite(s), preferably zeolite A, zeolite P, zeolite X and mixtures thereof, in amounts of from 10 to 60% by weight, preferably from 15 to 50% by weight, and in particular from 20 to 40% by weight, based in each case on the mass.

The particle sizes of the faujasite-type zeolites used with preference in the process of the invention is

preferably within the range from 0.1 up to 100 μm , more preferably between 0.5 and 50 μm , and in particular between 1 and 30 μm , in each case measured with standard particle size determination methods.

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It is generally preferred in this context to use finely divided solids in the masses for processing in accordance with the invention, irrespective of whether said solids comprise the abovementioned zeolites or other builders or bleaches, bleach activators or other solids. Very generally, preference is given to process variants wherein the average particle size of the solids used in the deformable mass(es) is below 400 μm , preferably below 300 μm , and in particular below 200 μm .

average particle size The in this case arithmetic mean of the individual particle sizes, which may vary. Particularly preferred processes are those wherein less than 10% by weight, preferably less than 5% by weight, and in particular less than 1% by weight, of the solids used in the deformable mass(es) have particle sizes above 1000 µm. The upper particle size range may be narrowed even further, so that particularly preferred processes are those wherein less than 15% by weight, preferably less than 10% by weight, and in particular less than 5% by weight, of the solids used in the deformable mass(es) have particle sizes above 800 µm.

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In general, however, even narrower particle size distributions are preferred, where the breadth of fluctuation about the average particle size is not more than 50%, preferably not more than 40%, and in particular not more than 30%, of the average particle size; i.e., the particle sizes make up at least 0.7 times and at most 1.3 times the average particle size.

Above, the weight ratio of water to certain ingredients in masses preferred in accordance with the invention for processing has been specified. After processing, this water is preferably bound in the form of hydrate water, so that the process end products preferably have a significantly lower free water content. Preferred end products of the process of the invention are essentially water-free; i.e., in a state in which the amount of liquid water, i.e., water not present in the form of hydrate water and/or constitution water, is less than 2% by weight, preferably less than 1% by weight, and in particular even below 0.5% by weight, based in each case on the tablets. Accordingly, preferred processes of the invention are those wherein the tablets comprise less than 10% by weight, preferably less than 5% by weight, with particular preference less than 1% by weight, and in particular less than 0.5% by weight, of free water. Water may accordingly be present essentially only in chemically and/or physically bound form or as a constituent of the solid raw materials or compounds, but not as a liquid, solvent or dispersion, in the end products of the process of the invention. Advantageously, the tablets at the end of the production process of the invention have an overall water content of not more than 15% by weight, with this water, therefore, being present not in liquid, free form but instead in chemically and/or physically bound form, and it is particularly preferred for the amount of water that is not bound to zeolite and/or silicates in the solid premix to amount to not more than 10% by weight and in particular not more than 7% by weight.

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Process end products which are particularly preferred
in the context of the present invention not only
possess an extremely small proportion of free water but
are preferably themselves still able to bind further
free water. In preferred processes, the water content

of the tablets is from 50 to 100% of the calculated water binding capacity.

The water binding capacity is the ability of a substance (in this case, of the process end product) to absorb water in chemically stable form, and ultimately indicates the amount of water which can be bound in the form of stable hydrates by a substance or by a tablet. The dimensionless value of the water binding capacity (WBC) is calculated from:

$$WBC = \frac{n \cdot 18}{M} ,$$

where n is the number of water molecules in the corresponding hydrate of the substance and M is the molar mass of the unhydrated substance. For the water binding capacity of anhydrous sodium carbonate (formation of sodium carbonate monohydrate), for example, this gives a value of

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$$WBC = \frac{1.18}{2 \cdot 23 + 12 + 3 \cdot 16} = 0.17.$$

The value WBC may be calculated for all hydrate-forming substances that are used in the masses for processing in accordance with the invention. The percentage proportions of these substances then produce the overall water binding capacity of the formulation. In preferred process end products, then, the water content is between 50 and 100% of this calculated value.

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In addition to the water content of the process end products and the ratio of water to certain raw materials, it is also to make statements about the absolute water content of the masses for processing in accordance with the invention. In particularly preferred processes, the deformable mass(es) in the course of

processing has (have) a water content of from 2.5 to 30% by weight, preferably from 5 to 25% by weight, and in particular from 7.5 to 20% by weight, based in each case on the mass.

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A further mechanism for the hardening of the masses processed in the process of the invention consists in the cooling of the masses during processing above their softening point. Processes wherein the hardening of the deformable mass(es) takes place by cooling below the melting point, accordingly, are preferred.

Masses which may be softened under the action of temperature may easily be formulated by mixing the desired further ingredients with a meltable or softenable substance, and heating the mixture to temperatures within the softening range of this substance and shaping the mixture at these temperatures. It is particularly preferred in this case to use waxes, paraffins, polyalkylene glycols, etc., as meltable or softenable substances. These substances are described below.

The meltable or softenable substances should have a melting range (solidification range) situated within a temperature range in which the other ingredients of the masses to be processed are not exposed to any excessive thermal load. On the other hand, however, the melting range must be sufficiently high still to provide a handleable tablet at at least slightly elevated temperature. In masses preferred in accordance with the invention, the meltable or softenable substances have a melting point above 30°C.

35 It has proven advantageous for the meltable or softenable substances not to exhibit a sharply defined melting point, as encountered commonly with pure, crystalline substances, but instead to have a melting

range which covers, in some cases, several degrees Celsius. The meltable or softenable substances preferably have a melting range which lies between about 45°C and above 75°C. In the present case that means that the melting range occurs within the stated temperature interval, and does not denote the width of the melting range. The width of the melting range is preferably at least 1°C, more preferably from about 2 to about 3°C.

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The abovementioned properties are in general possessed by what are called waxes. The term "waxes" is applied to a range of natural or synthetic substances which melt without decomposition, generally at above 40°C, and are of comparatively low viscosity, without stringing, at just a little above the melting point. They have a highly temperature-dependent consistency and solubility.

20 According to their origin, the waxes are divided into three groups: the natural waxes, chemically modified waxes, and the synthetic waxes.

The natural waxes include, for example, plant waxes 25 such as candelilla wax, carnauba wax, japan wax, esparto grass wax, cork wax, quaruma wax, rice germ oil wax, sugarcane wax, ouricury wax, or montan wax, animal waxes such as beeswax, shellac wax, spermaceti, lanolin (wool wax), uropygial grease, mineral waxes such as ceresin or ozokerite (earth wax), or petrochemical 30 waxes such petrolatum, paraffin as waxes microcrystalline waxes.

The chemically modified waxes include, for example, 35 hard waxes such as montan ester waxes, sassol waxes, or hydrogenated jojoba waxes.

By synthetic waxes are meant, in general, polyalkylene waxes or polyalkylene glycol waxes. As meltable or softenable substances for the masses which harden by cooling it is also possible to use compounds from other classes of substance which meet the stated requirements in terms of softening point. Examples of synthetic compounds which have proven suitable are higher esters of phthalic acid, especially dicyclohexyl phthalate, which is available commercially under the name Unimoll® 66 (Bayer AG). Also suitable are synthetically prepared waxes from lower carboxylic acids and fatty alcohols, example being dimyristyl tartrate. available under the name Cosmacol® ETLP (Condea). Conversely, synthetic or partially synthetic esters of lower alcohols with fatty acids from natural sources may also be used. This class of substance includes, for example, Tegin[®] 90 (Goldschmidt), a glyceryl monostearate palmitate. Shellac as well, for example, Schellack-KPS-Dreiring-SP (Kalkhoff GmbH), may be used according to the invention as meltable or softenable substances.

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Likewise counted among the waxes in the context of the present invention are, for example, the so-called wax alcohols. Wax alcohols are relatively high molecular mass, water-insoluble fatty alcohols having in general from about 22 to 40 carbon atoms. The wax alcohols occur, for example, in the form of wax esters of relatively high molecular mass fatty acids (wax acids) as a principal constituent of many natural waxes. Examples of wax alcohols are lignoceryl alcohol (1tetracosanol), cetyl alcohol, myristyl alcohol, melissyl alcohol. The coating of the particulate solids coated in accordance with the invention may, desired, also include wool wax alcohols, by which are meant triterpenoid and steroid alcohols, an example being lanolin, which is available under the commercial designation Argowax® (Pamentier & Co.), for example.

Likewise possible for use, at least proportionally, as a constituent of the coating are, in the context of the present invention, fatty acid glycerol esters or fatty acid alkanolamides, and also, if desired, water-insoluble or only sparingly water-soluble polyalkylene glycol compounds.

Particularly preferred meltable or softenable substances in the masses for processing are those from the group consisting of polyethylene glycols (PEG) and/or polypropylene glycols (PPG), preference being given to polyethylene glycols having molecular masses of between 1500 and 36,000, particular preference to those having molecular masses of from 2000 to 6000, and special preference to those having molecular masses of from 3000 to 5000. Corresponding processes wherein the plastically deformable mass(es) comprise(s) at least one substance from the group of polyethylene glycols and/or polypropylene glycols (PPG) are also preferred.

In this context, particularly preferred masses for processing in accordance with the invention are those comprising polypropylene glycols (PPG) polyethylene qlycols (PEG) as sole meltable softenable substances. Polypropylene (abbreviation PPG) which may be used in accordance with the invention are polymers of propylene glycol which satisfy the general formula I

$$H$$
-(O-CH-CH₂)_n-OH (I)
 CH_3

where n can adopt values between 10 and 2000. Preferred PPGs have molecular masses of between 1000 and 10,000

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and, correspondingly, values of n of between 17 and approximately 170.

Polyethylene glycols (abbreviation PEG) which may be used with preference in accordance with the invention are polymers of ethylene glycol which satisfy the general formula II

$$H-(O-CH_2-CH_2)_n-OH$$
 (II)

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where n can adopt values between 20 and approximately 1000. The abovementioned preferred molecular weight ranges correspond in this case to preferred ranges for the value n in formula II of from about 30 to about 820 (to be exact: from 34 to 818), with particular preference from about 40 to about 150 (to be exact: from 45 to 136), and in particular from about 70 to about 120 (to be exact: from 68 to 113).

- 20 another preferred embodiment, the masses processing in accordance with the invention comprise paraffin wax in the predominant fraction. That means that at least 50% by weight of the total meltable or softenable substances present, preferably more, consist 25 of paraffin wax. Particularly suitable are paraffin wax total meltable contents (based on or softenable approximately substances) of 60% by weight, approximately 70% by weight or approximately 80% by weight, with special preference being given to even higher proportions, of, for example, more than 90% by weight. In one particular embodiment of the invention, the total amount of the meltable or softenable substances used consists exclusively of paraffin wax.
- 35 Relative to the other, natural waxes mentioned, paraffin waxes have the advantage in the context of the present invention that in an alkaline cleaning product environment there is no hydrolysis of the waxes (as is

to be expected, for example, with the wax esters), since paraffin wax contains no hydrolyzable groups.

Paraffin waxes consist primarily of alkanes, plus low fractions of isoalkanes and cycloalkanes. The paraffin for use in accordance with the invention preferably contains essentially no constituents having a melting point of more than 70°C, with particular preference of more than 60°C. Below this melting temperature in the cleaning product liquor, fractions of high-melting alkanes in the paraffin may leave unwanted wax residues on the surfaces to be cleaned or on the ware to be cleaned. Wax residues of this kind lead in general to an unattractive appearance of the cleaned surface and should therefore be avoided.

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Masses for processing with preference comprise as meltable or softenable substances at least one paraffin wax having a melting range from 50°C to 60°C, preferred processes being those wherein deformable mass(es) comprise(s) a paraffin wax having a melting range of from 50°C to 55°C.

Preferably, the amount of alkanes, isoalkanes cycloalkanes which are solid at ambient temperature (generally from about 10 to about 30°C) in the paraffin wax used is as high as possible. The larger the amount of solid wax constituents in a wax at room temperature, the more useful that wax is in the context of the 30 present invention. As the proportion of solid wax constituents increases, there is an increase in the resistance of the process end products to impacts or friction on other surfaces, resulting in a longerlasting protection. High proportions of oils or liquid 35 wax constituents may weaken the tablets or tablet regions, as a result of which pores are opened and the active substances are exposed to the ambient influences mentioned at the outset.

In addition to paraffin as main constituent, the meltable or softenable substances may further comprise one or more of the abovementioned waxes or waxlike substances. In a further preferred embodiment of the present invention, the mixture forming the meltable or softenable substances should be such that the mass and the tablets or tablet constituent formed from it are at least substantially water-insoluble. At a temperature of about 30°C, the solubility in water should not exceed about 10 mg/l and preferably should be below 5 mg/l.

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In such cases, however, the meltable or softenable substances should have as low a solubility in water as possible, even in water at elevated temperature, in order as far as possible to avoid temperature-independent release of the active substances.

The principle described above is used for the delayed release of ingredients at a particular point in time in the cleaning operation and can be employed with particular advantage if rinsing is carried out in the main rinse cycle at a relatively low temperature (for example, 55°C), so that the active substance is not released from the rinse aid particles until the rinse cycle at higher temperatures (approximately 70°C).

Preferred masses to be processed in accordance with the invention comprise as meltable or softenable substances one or more substances having a melting range of from 40°C to 75°C in amounts of from 6 to 30% by weight, preferably from 7.5 to 25% by weight, and in particular from 10 to 20% by weight, based in each case on the weight of the mass.

A further mechanism by which the masses may harden is the evaporation of solvents. To this end it is possible

to prepare solutions or dispersions of the desired ingredients in one or more suitable, volatile solvents, said solutions or dispersions emitting this (these) solvent(s) after the shaping step and, in so doing, hardening. Examples of appropriate solvents are lower alkanols, aldehydes, ethers, esters, etc., which are selected in accordance with the further composition of the masses for processing. Particularly suitable solvents for such processes wherein the deformable mass(es) harden(s) by evaporation of solvents ethanol, propanol, isopropanol, 1-butanol, 2-butanol, 2-methyl-1-propanol, 2-methyl-2-propanol, 1-pentanol, 2-pentanol, 3-pentanol, 2,2-dimethyl-1-propanol, 3-methyl-2-butanol, 3-methyl-1-butanol, 2-methyl-2butanol, 2-methyl-1-butanol, 1-hexanol, and acetates of the abovementioned alcohols, especially ethyl acetate.

The evaporation of the abovementioned solvents may be accelerated by heating after shaping and cutting to length, or by air movement. Combinations of the measures specified are also suitable for this purpose, for example, the blowing of the cut-to-length tablets with warm or hot air.

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A further mechanism which may form the basis for the hardening of the shaped and cut-to-length masses is that of crystallization. Processes wherein the deformable mass(es) harden(s) by crystallization are likewise preferred.

Crystallization, as a mechanism on which the hardening is based, may be utilized by using, for example, melts of crystalline substances as the basis of one or more shapable masses. Following processing, systems of this kind undergo transition to a higher state of order, which in turn leads to hardening of the overall tablet formed. Alternatively, crystallization may take place

by crystallization from supersaturated solution. In the invention, context of the present supersaturation refers to a metastable state in which, in a closed more of one substance is present than is required for saturation. A supersaturated solution obtained, for example, by undercooling accordingly comprises more dissolved substance than it should contain in thermal equilibrium. The excess of dissolved substance may be brought to instantaneous crystallization by seeding with seed crystal or dust particles or by agitating the system. In the context of the present invention, the term "supersaturated" always refers to a temperature of 20°C. If x grams of a substance per liter dissolve in a defined solvent at a temperature of 20°C, then the solution, in the context of the present invention, may be referred to as "supersaturated" if it contains (x + y) grams of the substance per liter, y being > 0. Consequently, in the context of the present invention, solutions referred to as "supersaturated" include those which at an elevated temperature are used as the basis of a mass for processing and are processed at this temperature, in which more dissolved substance is present in the solution than would dissolve in the same amount of solvent at 20°C.

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The term "solubility" is understood by the present invention to mean the maximum amount of a substance which the solvent is able to accommodate at a certain temperature, i.e., the fraction of the substance in a solution saturated at the temperature in question. Where a solution contains more dissolved substance than it should contain in thermodynamic equilibrium at a given temperature (for example, in the case of undercooling), it is referred to as supersaturated. By seeding with seed crystals it is possible to cause excess to precipitate as a sediment solution, which is now just saturated. A solution saturated in respect of a substance may, however, also dissolve other substances (for example, it is still possible to dissolve sugar in a saturated solution of common salt).

state of supersaturation can be achieved, 5 described above, by slow cooling or by undercooling of a solution, provided the dissolved substance is more soluble in the solvent at higher temperatures. Other possibilities to obtain supersaturated solutions are, for example, the combination of two solutions whose 10 ingredients react to form another substance which does (hindered or retarded immediately precipitate The latter mechanism precipitation reactions). particularly suitable as a basis for the formation of masses for processing in accordance with the invention. 15

In principle, the state of supersaturation is achievable in any kind of solution, although the principle described in the present specification finds application, as already mentioned, in the production of laundry detergents and cleaning products. Accordingly, some systems, which in principle tend to form supersaturated solutions, are less suitable for use in accordance with the invention, since the systems of substance on which they are based cannot be used, on environmental, toxicological, or economic grounds. In addition to nonionic surfactants or common nonaqueous solvents, therefore, particular preference is given to processes of the invention with the last-mentioned hardening mechanism wherein a supersaturated aqueous solution is used as the basis of at least one mass for processing.

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As already mentioned above, the state of supersaturation in the context of the present invention refers to the saturated solution at 20°C. By using solutions which have a temperature above 20°C it is easy to attain the state of supersaturation. Process of the invention wherein the crystallization-hardening mass during processing has a temperature of between 35 and 120°C, preferably between 40 and 110°C, with particular preference between 45 and 90°C, and in particular between 50 and 80°C, are preferred in the context of the present invention.

laundry detergent and cleaning product the tablets produced are generally neither stored elevated temperatures nor later used at these elevated temperatures, the cooling of the mixture leads to the precipitation from the supersaturated solution of the fraction of dissolved substance which was present in the solution above the saturation limit at 20°C. Thus, on cooling, the supersaturated solution may divided into a saturated solution and a sediment. An alternative possibility is that, owing to recrystallization and hydration phenomena, the supersaturated solution solidifies on cooling to form a solid. This is the certain salts example, if containing case, for their water of dissolve in hydration water this heating. In case, crystallization on supersaturated solutions are often formed on cooling which by mechanical action or addition of seed crystal solid the salt, containing water of which is crystallization, the state as room temperature thermodynamically stable at solidify. This phenomenon is known, for example, for sodium thiosulfate pentahydrate and sodium acetate trihydrate, the latter salt in particular, containing hydration water, being suitable for use with advantage in the form of the supersaturated solution in the process of the invention. Specific laundry detergent and cleaning product ingredients as well, phosphonates, for example, display this phenomenon and are outstandingly suitable in the form of the solutions as granulation aids. For this purpose the corresponding phosphonic acids (see below) are neutralized with

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concentrated alkali, the solution becoming heated as a result of the heat of neutralization. On cooling, these solutions form solids of the corresponding alkali metal phosphonates. By incorporating further laundry deterproduct ingredients gent and cleaning solutions while still hot, it is possible in accordance with the invention to prepare processable masses of different composition. Particularly preferred processes of the invention are those wherein the supersaturated solution used as a basis of the hardening solidifies at room temperature to form a solid. It is preferred in this case that the formerly supersaturated solution, following solidification to form a solid, cannot be converted back into a supersaturated solution by heating to the temperature at which the supersaturated solution was formed. This is the case, for example, with the phosphonates mentioned.

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As mentioned above, the supersaturated solution used as a basis of the hardening mass may be obtained in a 20 number of ways and then processed in accordance with the invention following optional admixing of further ingredients. One simple way, for example, is to prepare the supersaturated solution which is used as the basis of the hardening mass by dissolving the dissolved sub-25 stance in heated solvent. If the amounts of dissolved substance that are dissolved in this way in the heated solvent are higher than those which would dissolve at 20°C, then a solution is present which is supersaturated within the meaning of the present 30 invention and which either hot (see above) or after cooling, and in the metastable state, may be introduced into the mixer.

A further possibility is to remove the water from salts containing hydration water by "dry" heating and to dissolve them in their own water of crystallization (see above). This too is a method of preparing super-

saturated solutions that may be used in the context of the present invention.

Another way is to add a gas or other fluid or solution to a non-supersaturated solution, so that the dissolved substance reacts in the solution to form a less soluble substance or dissolved to a lesser extent mixture of the solvents. The combination solutions each containing two substances which react with one another to form a less soluble substance is likewise a method of preparing supersaturated solutions, provided the less-soluble substance does not precipitate instantaneously. Processes which are likewise preferred in the context of the present invention are those wherein the supersaturated solution used as the basis of the hardening mass is prepared by combining two or more solutions. Examples of such ways of preparing supersaturated solutions are dealt with below.

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Preferred processes of the invention are those wherein the supersaturated aqueous solution is obtained by combining an aqueous solution of one or more acidic ingredients of laundry detergents and cleaning products, preferably from the group of the surfactant acids, the builder acids, and the complexing agent acids, and an aqueous alkali solution, preferably an aqueous alkali metal hydroxide solution, in particular an aqueous sodium hydroxide solution.

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Among the representatives of said classes of compound that have already been mentioned earlier on above, the phosphonates in particular occupy an outstanding position in the context of the present invention. In preferred processes of the invention, therefore, the supersaturated aqueous solution is obtained by combining an aqueous phosphonic acid solution with concentrations above 45% by weight, preferably above 50% by

weight, and in particular above 55% by weight, based in each case on the phosphonic acid solution, and an aqueous sodium hydroxide solution with concentrations above 35% by weight, preferably above 40% by weight, and in particular above 45% by weight, based in each case on the sodium hydroxide solution.

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The hardening of the deformable mass(es) may, in accordance with the invention, also take place by means of chemical reaction(s), especially addition polymeri-10 zation. Suitable in this context, in principle, are all chemical reactions which, starting from one or more liquid to paste-like substances, lead, by reaction with (an) other substance(s), to solids. Especially suitable 15 in this context are chemical reactions which do not lead suddenly to said change of state. From the multitude of chemical reactions which lead to solidification phenomena, suitable reactions are in particular those in which larger molecules are built up from smaller 20 molecules. These reactions include, in turn, preferably reactions in which many small molecules react to form (one) larger molecule(s). These are what are known as polymerization reactions (polyaddition, addition polymerization, polycondensation) and polymer-analogous 25 reactions. The corresponding addition polyadducts (poly-addition products) or polycondensates (polycondensation products) then give the finished, cut-to-length tablet its strength.

In view of the intended use of the products produced in accordance with the invention it is preferred to utilize as hardening mechanism the formation of those solid substances from liquid or paste-like starting materials which are in any case to be used in the laundry detergent and cleaning product as ingredients, examples being cobuilders, soil repellents, and soil release polymers. Such cobuilders may originate, for example, from the groups of the polycarboxylates/poly-

carboxylic acids, polymeric polycarboxylates, aspartic acid, polyacetals, dextrins, etc. These classes of substance are described later on.

- A further mechanism by which the deformable mass(es) may harden in the context of the present invention is that of hardening as a result of a change in rheological properties.
- In this case, use is made of the property possessed by certain substances of changing in some instances, drastically their rheological properties under the action of shear forces. Examples of such systems, which are familiar to the skilled worker, are phyllosilicates, for example, which under shearing have a highly thickening action in appropriate matrices and may lead to masses of firm consistency.

It is of course possible for two or more hardening mechanisms to be combined with one another and/or used simultaneously in one mass. Appropriate in this case, for example, are crystallization with simultaneous solvent evaporation, cooling with simultaneous crystallization, water binding ("internal drying") with simultaneous external drying, etc.

The general description of mechanisms which may form the basis for hardening in the process of the invention is followed by a detailed description of the other ingredients to be used in the masses for processing.

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Preferred end products of the process of the invention, i.e. preferred laundry detergent and cleaning product tablets, further comprise one or more surfactants. Accordingly, it is preferred for at least one of the masses for processing to comprise surfactant(s). In the laundry detergent and cleaning product tablets of the invention it is possible to use anionic, nonionic,

cationic and/or amphoteric surfactants, and/or mixtures thereof. From a performance standpoint, preference is given to mixtures of anionic and nonionic surfactants. The total surfactant content of the tablets is for laundry detergent tablets from 5 to 60% by weight, based on the tablet weight, preference being given to surfactant contents of more than 15% by weight, while for cleaning product tablets machine dishwashing contain preferably less than 5% by weight surfactant(s).

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Anionic surfactants used are, for example, those of the sulfonate and sulfate type. Preferred surfactants of the sulfonate type are C9-13 alkylbenzenesulfonates, olefinsulfonates, i.e., mixtures of alkenesulfonates and hydroxyalkanesulfonates, and also disulfonates, as are obtained, for example, from C₁₂₋₁₈ monoolefins having a terminal or internal double bond by sulfonating with gaseous sulfur trioxide followed by alkaline or acidic 20 hydrolysis of the sulfonation products. Also suitable are alkanesulfonates, which are obtained from C12-18 alkanes, example, by sulfochlorination sulfoxidation with subsequent hydrolysis or neutralization, respectively. Likewise suitable, addition, are the esters of α -sulfo fatty acids (ester sulfonates), e.g., the α -sulfonated methyl esters of hydrogenated coconut, palm kernel or tallow fatty acids.

Further suitable anionic surfactants are sulfated fatty acid glycerol esters. Fatty acid glycerol esters are the monoesters, diesters and triesters, and mixtures thereof. as obtained in the preparation bv esterification of a monoglycerol with from 1 to 3 mol 35 fatty acid or in the transesterification triglycerides with from 0.3 to 2 mol of glycerol. Preferred sulfated fatty acid glycerol esters are the sulfation products of saturated fatty acids having 6 to

22 carbon atoms, examples being those of caproic acid, caprylic acid, capric acid, myristic acid, lauric acid, palmitic acid, stearic acid, or behenic acid.

Preferred alk(en)yl sulfates are the alkali metal salts, and especially the sodium salts, of the sulfuric monoesters of $C_{12}\text{-}C_{18}$ fatty alcohols, examples being those of coconut fatty alcohol, tallow fatty alcohol, lauryl, myristyl, cetyl or stearyl alcohol, or of $C_{10}\mbox{-}C_{20}$ monoesters of secondary those alcohols, and 10 alcohols of these chain lengths. Preference is also given to alk(en)yl sulfates of said chain length which contain a synthetic straight-chain alkyl radical prepared on a petrochemical basis, these sulfates possessing degradation properties similar to those of 15 the corresponding compounds based on fatty-chemical raw materials. From a detergents standpoint, the $C_{12}\text{-}C_{16}$ alkyl sulfates and $C_{12}\text{-}C_{15}$ alkyl sulfates, and also C_{14} - C_{15} alkyl sulfates, are preferred. In addition, 2,3alkyl sulfates, which may for example be prepared in 20 accordance with US Patents 3,234,258 or 5,075,041 and obtained as commercial products from Shell Oil Company under the name DAN®, are suitable anionic surfactants.

Also suitable are the sulfuric monoesters of the straight-chain or branched C₇₋₂₁ alcohols ethoxylated with from 1 to 6 mol of ethylene oxide, such as 2-methyl-branched C₉₋₁₁ alcohols containing on average 3.5 mol of ethylene oxide (EO) or C₁₂₋₁₈ fatty alcohols containing from 1 to 4 EO. Because of their high foaming behavior they are used in cleaning products only in relatively small amounts, for example, in amounts of from 1 to 5% by weight.

35 Further suitable anionic surfactants include the salts of alkylsulfosuccinic acid, which are also referred to as sulfosuccinates or as sulfosuccinic esters and which constitute monoesters and/or diesters of sulfosuccinic

acid with alcohols, preferably fatty alcohols and fatty alcohols. especially ethoxylated sulfosuccinates comprise C_{8-18} fatty alcohol radicals or mixtures thereof. Especially preferred sulfosuccinates derived radical fatty alcohol contain a ethoxylated fatty alcohols which themselves represent see below). nonionic surfactants (for description, turn is given in preference Particular whose fatty alcohol radicals sulfosuccinates derived from ethoxylated fatty alcohols having narrowed homolog distribution. Similarly, it is also possible to use alk(en)ylsuccinic acid containing preferably 8 to 18 carbon atoms in the alk(en)yl chain, or salts thereof.

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suitable anionic surfactants are, Further particular, soaps. Suitable soaps include saturated fatty acid soaps, such as the salts of lauric acid, stearic acid, myristic acid, palmitic hydrogenated erucic acid and behenic acid, and, 20 particular, mixtures of soaps derived from natural fatty acids, e.g., coconut, palm kernel, or tallow fatty acids.

The anionic surfactants, including the soaps, may be present in the form of their sodium, potassium or ammonium salts and also as soluble salts of organic bases, such as mono-, di- or triethanolamine. Preferably, the anionic surfactants are in the form of their sodium or potassium salts, in particular in the form of the sodium salts.

Nonionic surfactants used are preferably alkoxylated, advantageously ethoxylated, especially primary, alcohols having preferably 8 to 18 carbon atoms and on average from 1 to 12 mol of ethylene oxide (EO) per mole of alcohol, in which the alcohol radical may be linear or, preferably, methyl-branched in position 2

and/or may comprise linear and methyl-branched radicals in a mixture, as are commonly present in oxo alcohol radicals. In particular, however, preference is given to alcohol ethoxylates containing linear radicals from alcohols of natural origin having 12 to 18 carbon atoms, e.g., from coconut, palm, tallow fatty or oleyl alcohol, and on average from 2 to 8 EO per mole of alcohol. Preferred ethoxylated alcohols include, example, C_{12-14} alcohols containing 3 EO or 4 EO, C_{9-11} alcohol containing 7 EO, C₁₃₋₁₅ alcohols containing 3 EO, 5 EO, 7 EO or 8 EO, C_{12-18} alcohols containing 3 EO, 5 EO or 7 EO, and mixtures thereof, such as mixtures of C_{12-14} alcohol containing 3 EO and C_{12-18} alcohol containing The stated degrees of ethoxylation represent statistical mean values, which for a specific product may be an integer or a fraction. Preferred alcohol distribution ethoxylates have a narrowed homolog (narrow range ethoxylates, NREs). In addition to these nonionic surfactants it is also possible to use fatty alcohols containing more than 12 EO. Examples thereof are tallow fatty alcohol containing 14 EO, 25 EO, 30 EO or 40 EO.

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As further nonionic surfactants, furthermore, use may also be made of alkyl glycosides of the general formula 25 $RO(G)_x$, where R is a primary straight-chain or methylbranched aliphatic radical, especially an aliphatic radical methyl-branched in position 2, containing 8 to 22, preferably 12 to 18, carbon atoms, and G is the symbol representing a glycose unit having 5 or 6 carbon 30 preferably qlucose. The degree oligomerization, x, which indicates the distribution of monoglycosides and oligoglycosides, is any desired number between 1 and 10; preferably, x is from 1.2 to 35 1.4.

A further class of nonionic surfactants used with preference, which are used either as sole nonionic

surfactant or in combination with other nonionic surfactants, are alkoxylated, preferably ethoxylated, or ethoxylated and propoxylated, fatty acid alkyl esters, preferably having 1 to 4 carbon atoms in the alkyl chain, especially fatty acid methyl esters, as are described, for example, in Japanese Patent Application JP 58/217598, or those prepared preferably by the process described in International Patent Application WO-A-90/13533.

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Nonionic surfactants of the amine oxide type, examples being N-cocoalkyl-N,N-dimethylamine oxide and N-tallowalkyl-N,N-dihydroxyethylamine oxide, and of the fatty acid alkanolamide type, may also be suitable. The amount of these nonionic surfactants is preferably not more than that of the ethoxylated fatty alcohols, in particular not more than half thereof.

Further suitable surfactants are polyhydroxy fatty acid amides of the formula (III),

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|
R-CO-N-[Z] (III)

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where RCO is an aliphatic acyl radical having 6 to 22 an alkyl atoms, R^1 is hydrogen orcarbon hydroxyalkyl radical having 1 to 4 carbon atoms, and is a linear or branched polyhydroxyalkyl radical having 3 to 10 carbon atoms and from 3 to 10 hydroxyl groups. The polyhydroxy fatty acid amides are known obtainable by substances are customarily which reductive amination of a reducing sugar with ammonia, alkylamine or an alkanolamine, and subsequent acylation with a fatty acid, a fatty acid alkyl ester or a fatty acid chloride.

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The group of the polyhydroxy fatty acid amides also includes compounds of the formula (IV)

 R^1-O-R^2 | R-CO-N-[Z] (IV)

where R is a linear or branched alkyl or alkenyl radical having 7 to 12 carbon atoms, R¹ is a linear, branched or cyclic alkyl radical or an aryl radical having 2 to 8 carbon atoms and R² is a linear, branched or cyclic alkyl radical or an aryl radical or an oxyalkyl radical having 1 to 8 carbon atoms, preference being given to C₁-4 alkyl radicals or phenyl radicals, and [Z] is a linear polyhydroxyalkyl radical whose alkyl chain is substituted by at least two hydroxyl groups, or alkoxylated, preferably ethoxylated or propoxylated, derivatives of said radical.

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[Z] is preferably obtained by reductive amination of a reduced sugar, e.g., glucose, fructose, maltose, lactose, galactose, mannose, or xylose. The N-alkoxyor N-aryloxy-substituted compounds may then be converted to the desired polyhydroxy fatty acid amides, for example, in accordance with the teaching of International Patent Application WO-A-95/07331 by reaction with fatty acid methyl esters in the presence of an alkoxide as catalyst.

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In the context of the present invention, preference is given to producing laundry detergent and cleaning product tablets comprising anionic and nonionic surfactant(s); performance advantages may result from certain proportions in which the individual classes of surfactant are used.

For example, particular preference is given to laundry detergent and cleaning product tablets in which the surfactant(s) nonionic of anionic is between 10:1 and 1:10, preferably surfactant(s) between 7.5:1 and 1:5, and in particular between 5:1 1:2. Also preferred are laundry detergent tablets comprising surfactant(s), cleaning product preferably anionic and/or nonionic surfactant(s), in amounts of from 5 to 40% by weight, preferably from 7.5 to 35% by weight, with particular preference from 10 to 30% by weight, and in particular from 12.5 to 25% by weight, based in each case on the tablet weight.

From a performance standpoint it may be advantageous if certain classes of surfactant are absent from some phases of the laundry detergent and cleaning product tablets or from the tablet as a whole, i.e., from all phases. A further important embodiment of the present invention therefore envisages that at least one phase of the tablets is free from nonionic surfactants.

Conversely, however, the presence of certain surfactants in individual phases or in the whole tablet, i.e., in all phases, may produce a positive effect. The incorporation of the above-described alkyl polyglycosides has been found advantageous, and so preference is given to laundry detergent and cleaning product tablets in which at least one phase of the tablets comprises alkyl polyglycosides.

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Similarly to the case with the nonionic surfactants, the omission of anionic surfactants from certain phases or all phases may also result in laundry detergent and cleaning product tablets better suited to certain fields of application. In the context of the present invention, therefore, it is also possible to conceive of laundry detergent and cleaning product tablets in

which at least one phase of the tablet is free from anionic surfactants.

As already mentioned, the use of surfactants in the product tablets for machine cleaning of the use preferably limited to dishwashing is amounts. Laundry small nonionic surfactants in detergent and cleaning product tablets preferred for use as cleaning product tablets in the context of the present invention are those which have total surfactant 10 contents of less than 5% by weight, preferably less than 4% by weight, with particular preference less than 3% by weight, and in particular less than 2% by weight, based in each case on their total weight. Surfactants used in machine dishwashing compositions are usually 15 only low-foaming nonionic surfactants. Representatives from the groups of the anionic, cationic and amphoteric surfactants, in contrast, are of relatively little importance. With particular preference, the cleaning product tablets produced according to the invention for 20 machine dishwashing comprise nonionic surfactants, especially nonionic surfactants from the group of the alkoxylated alcohols. Preferred nonionic surfactants advantageously ethoxylated, alkoxylated, are especially primary, alcohols having preferably 8 to 18 25 carbon atoms and on average from 1 to 12 mol of ethylene oxide (EO) per mole of alcohol, in which the alcohol radical may be linear or, preferably, methylbranched in position 2 and/or may contain a mixture of linear and methyl-branched radicals, as are customarily 30 present in oxo alcohol radicals. Particular preference is given, however, to alcohol ethoxylates having linear radicals from alcohols of natural origin having 12 to 18 carbon atoms, e.g., from coconut, palm, tallow fatty or oleyl alcohol, and having on average from 2 to 8 EO 35 per mole of alcohol. The preferred ethoxylated alcohols include, for example, C_{12-14} alcohols having 3 EO or 4 EO, C₉₋₁₁ alcohol having 7 EO, C₁₃₋₁₅ alcohols having

3 EO, 5 EO, 7 EO or 8 EO, C_{12-18} alcohols having 3 EO, 5 EO or 7 EO, and mixtures of these, such as mixtures of C_{12-14} alcohol having 3 EO and C_{12-18} alcohol having of ethoxylation stated degrees 5 EO. The statistical means, which for a specific product may be an integer or a fraction. Preferred alcohol ethoxylates have a narrowed homolog distribution (narrow range In addition to these nonionic NREs). ethoxylates, surfactants, fatty alcohols having more than 12 EO may also be used. Examples thereof are tallow fatty alcohol having 14 EO, 25 EO, 30 EO, or 40 EO.

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in connection with the production, Especially accordance with the invention, of laundry detergent or cleaning product tablets for 15 tablets dishwashing, it is preferred for the laundry detergent and cleaning product tablets to comprise a nonionic point above room a melting having surfactant least one Accordingly, at temperature. deformable masses in the process of the invention 20 preferably comprises a nonionic surfactant having a melting point above 20°C. Nonionic surfactants whose is preferred have melting points above 25°C, is particularly whose use nonionic surfactants preferred have melting points of between 25 and 60°C, 25 in particular between 26.6 and 43.3°C.

having melting surfactants nonionic Suitable softening points within the stated temperature range low-foaming nonionic for example, are. highly viscous at solid or may be temperature. If nonionic surfactants which are highly viscous at room temperature are used, then it preferred that they have a viscosity above 20 Pas, preferably above 35 Pas, and in particular above 40 Pas. Also preferred are nonionic surfactants which possess a waxlike consistency at room temperature.

Preferred nonionic surfactants for use that are solid room temperature originate from the groups especially surfactants, alkoxylated nonionic ethoxylated primary alcohols, and mixtures of these surfactants of more with surfactants construction such as polyoxypropylene/polyoxyethylene/ surfactants. (PO/EO/PO) polyoxypropylene notable, nonionic surfactants are (PO/EO/PO) furthermore, for good foam control.

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In one preferred embodiment of the present invention, the nonionic surfactant having a melting point above room temperature is an ethoxylated nonionic surfactant originating from the reaction of a monohydroxy alkanol alkylphenol having 6 to 20 carbon atoms with preferably at least 12 mol, with particular preference at least 15 mol, in particular at least 20 mol, of ethylene oxide per mole of alcohol or alkylphenol, respectively.

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alkylphenols,

A particularly preferred nonionic surfactant for use that is solid at room temperature is obtained from a straight-chain fatty alcohol having 16 to 20 carbon atoms (C_{16-20} alcohol), preferably a C_{18} alcohol, and at least 12 mol, preferably at least 15 mol, and in particular at least 20 mol of ethylene oxide. Of these, the so-called "narrow range ethoxylates" (see above) are particularly preferred.

solid surfactant which is at nonionic 30 temperature preferably further possesses propylene oxide units in the molecule. Preferably, such PO units account for up to 25% by weight, with particular preference up to 20% by weight, and in particular up to 15% by weight, of the overall molar mass of 35 nonionic surfactant. Particularly preferred nonionic surfactants are ethoxylated monohydroxy alkanols or additionally comprise

which.

polyoxyethylene-polyoxypropylene block copolymer units. The alcohol or alkylphenol moiety of such nonionic surfactant molecules in this case makes up preferably more than 30% by weight, with particular preference more than 50% by weight, and in particular more than 70% by weight, of the overall molar mass of such nonionic surfactants.

Further nonionic surfactants whose use is particularly preferred, have melting points above room temperature, 10 70% of a polyoxypropylene/ 40 to contain from polyoxyethylene/polyoxypropylene block polymer blend which comprises 75% by weight of an inverted block polyoxyethylene and polyoxypropylene of copolymer containing 17 mol of ethylene oxide and 44 mol of 15 propylene oxide and 25% by weight of a block copolymer of polyoxyethylene and polyoxypropylene, initiated with trimethylolpropane and containing 24 mol of ethylene oxide and 99 mol of propylene oxide per mole of trimethylolpropane. 20

Nonionic surfactants which may be used with particular preference are, for example, obtainable under the name Poly Tergent® SLF-18 from the company Olin Chemicals.

A further preferred surfactant may be described by the formula

 $R^{1}O[CH_{2}CH(CH_{3})O]_{x}[CH_{2}CH_{2}O]_{y}[CH_{2}CH(OH)R^{2}]$

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linear or branched aliphatic R^1 is a which hydrocarbon radical having 4 to 18 carbon atoms, or thereof, R^2 is a linear or branched mixtures hydrocarbon radical having 2 to 26 carbon atoms, or mixtures thereof, and x is between 0.5 and 1.5, and yis at least 15.

Further nonionic surfactants which may be used with preference are the endgroup-capped poly(oxyalkylated) nonionic surfactants of the formula

5 $R^{1}O[CH_{2}CH(R^{3})O]_{x}[CH_{2}]_{k}CH(OH)[CH_{2}]_{j}OR^{2}$

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in which R^1 and R^2 are linear or branched, saturated or unsaturated, aliphatic or aromatic hydrocarbon radicals having 1 to 30 carbon atoms, R^3 is H or a methyl, ethyl, n-propyl, isopropyl, n-butyl, 2-butyl or 2-10 methyl-2-butyl radical, x is between 1 and 30, k and j are between 1 and 12, preferably between 1 and 5. Where $x \ge 2$, each R^3 in the above formula may be different. R^1 and R² are preferably linear or branched, saturated or unsaturated, aliphatic or aromatic hydrocarbon radicals 15 having 6 to 22 carbon atoms, radicals having 8 to 18 carbon atoms being particularly preferred. For the or -CH₂CH₃ are particularly radical R³, Η, -CH₃ preferred. Particularly preferred values for x lie within the range from 1 to 20, in particular from 6 to 20 15.

As described above, each R^3 in the above formula may be different if $x \ge 2$. By this means it is possible to vary the alkylene oxide unit in the square brackets. If x, for example, is 3, the radical R^3 may be selected in order to form ethylene oxide $(R^3 = H)$, or propylene oxide $(R^3 = CH_3)$ units, which may be added on to one another in any sequence, examples being (EO) (PO) (EO) (EO

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Particularly preferred endgroup-capped poly(oxy-alkylated) alcohols of the above formula have values of k=1 and j=1, thereby simplifying the above formula to

 $R^{1}O[CH_{2}CH(R^{3})O]_{x}CH_{2}CH(OH)CH_{2}OR^{2}.$

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In the last-mentioned formula, R^1 , R^2 and R^3 are as defined above and x stands for numbers from 1 to 30, preferably from 1 to 20, and in particular from 6 to 18. Particular preference is given to surfactants wherein the radicals R^1 and R^2 have 9 to 14 carbon atoms, R^3 is H, and x adopts values from 6 to 15.

The remarks above refer in part to the process end 15 products, which - as mentioned earlier on - may also be or four-phase two-, threeform of individual mass Based on the configurations. processing, which comprises surfactant(s), preference in connection with the production of cleaning product 20 tablets for machine dishwashing is given to processes wherein the deformable mass(es) has (have) total than 5% by weight, surfactant contents of less preferably less than 4% by weight, with particular preference less than 3% by weight, and in particular less than 2% by weight, based in each case on the mass.

In addition to the abovementioned constituents, builder and surfactant, the laundry detergent and cleaning product tablets of the invention may comprise further customary laundry detergent and cleaning product ingredients from the group consisting of bleaches, disintegration aids, dyes, activators, bleach brighteners, enzymes, optical fragrances, silicone oils, antiredeposition agents, inhibitors, graying inhibitors, color transfer inhibitors, corrosion inhibitors. These substances may be used in all of the masses for processing, although it is also

possible to make use of advantageous properties by virtue of the separation of certain ingredients.

In order to facilitate the disintegration of highly is possible to incorporate compacted tablets, it disintegration aids, known as tablet disintegrants, into the tablets in order to reduce the disintegration times. These substances are suitable, for example, for accelerating the release of individual tablet regions In the process of relative to other regions. 10 invention, this may be realized in that only one of the masses for processing comprises such substances, or in that two or more masses comprise such substances in disintegrants, or Tablet amounts. different understood in disintegration accelerators, are accordance with Römpp (9th Edition, Vol. 6, p. 4440) and Voigt "Lehrbuch der pharmazeutischen Technologie" [Textbook of pharmaceutical technology] (6th Edition, 1987, pp. 182-184) to be auxiliaries which ensure the rapid disintegration of tablets in water or gastric 20 fluid and the release of the drugs in absorbable form.

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These substances increase in volume on ingress water, with on the one hand an increase intrinsic volume (swelling) and on the other hand, by way of the release of gases as well, the possiblity of generating a pressure which causes the tablets to smaller particles. Examples into disintegrate established disintegration aids are carbonate/citric acid systems, with the use of other organic acids also being possible. Examples of swelling disintegration such as synthetic polymers aids are polyvinylpyrrolidone (PVP) or natural polymers and/or modified natural substances such as cellulose and starch and their derivatives, alginates, or casein derivatives.

Preferred laundry detergent and cleaning product tablets contain from 0.5 to 10% by weight, preferably from 3 to 7% by weight, and in particular from 4 to 6% by weight, of one or more disintegration aids, based in each case on the tablet weight. If only one mass comprises disintegration aids, then these figures are based only on the weight of that mass.

Preferred disintegrants used in the context of the present invention are cellulose-based disintegrants and 10 so preferred laundry detergent and cleaning product tablets comprise a cellulose-based disintegrant of this kind in amounts from 0.5 to 10% by weight, preferably from 3 to 7% by weight, and in particular from 4 to 6% by weight. Pure cellulose has the formal empirical 15 composition $(C_6H_{10}O_5)_n$ and, considered formally, is a which itself of cellobiose, β -1,4-polyacetal constructed of two molecules of glucose. Suitable celluloses consist of from about 500 to 5000 glucose units and, accordingly, have average molecular masses 20 500,000. Cellulose-based to 50,000 of from disintegrants which can be used also include, in the context of the present invention, cellulose derivatives polymer-analogous reactions obtainable by cellulose. Such chemically modified celluloses include, 25 products esterifications of example, etherifications in which hydroxy hydrogen atoms have been substituted. However, celluloses in which the hydroxy groups have been replaced by functional groups not attached by an oxygen atom may also be used as The group of the cellulose cellulose derivatives. alkali metal embraces, for example, derivatives carboxymethylcellulose (CMC), cellulose celluloses, esters and cellulose ethers and aminocelluloses. Said cellulose derivatives are preferably not used alone as cellulose-based disintegrants but instead are used in a cellulose derivative The mixture with cellulose. content of these mixtures is preferably less than 50%

by weight, with particular preference less than 20% by weight, based on the cellulose-based disintegrant. The particularly preferred cellulose-based disintegrant used is pure cellulose, free from cellulose derivatives.

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The cellulose used as disintegration aid is preferably not used in finely divided form but instead a coarser form, for example, by into converted granulation or compaction, before being admixed to the premixes intended for compression. Laundry detergent and cleaning product tablets comprising disintegrants optionally cogranulated form granular orPatent Applications described German in DE 197 10 254 and Herzog) DE 197 09 991 (Stefan International Patent Application and in (Henkel) documents also provide (Henkel). These WO98/40463 granulated, further details on the production of compacted or cogranulated cellulose disintegrants. The particle sizes of such disintegrants are usually above 200 μm , preferably between 300 and 1600 μm to the extent of at least 90%, and in particular between 400 and 1200 μm to the extent of at least 90%. abovementioned, relatively coarse disintegration aids, and those described in more detail in the cited documents, are preferred for use as disintegration aids in the context of the present invention and example, under the commercially, for available from the company Arbocel® TF-30-HG designation Rettenmaier.

As a further cellulose-based disintegrant or as a constituent of this component it is possible to use microcrystalline cellulose. This microcrystalline cellulose is obtained by partial hydrolysis of celluloses under conditions which attack only the amorphous regions (approximately 30% of the total cellulose mass) of the celluloses and break them up

completely but leave the crystalline regions (approximately 70%) intact. Subsequent deaggregation of the microfine celluloses resulting from the hydrolysis yields the microcrystalline celluloses, which have primary particle sizes of approximately 5 μm and can be compacted, for example, to granules having an average particle size of 200 μm .

Laundry detergent and cleaning product tablets which are preferred in the context of the present invention further comprise a disintegration aid, preferably a cellulose-based disintegration aid, preferably in granular, cogranulated or compacted form, in amounts of from 0.5 to 10% by weight, preferably from 3 to 7% by weight, and in particular from 4 to 6% by weight, based in each case on the tablet weight.

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The laundry detergent and cleaning product tablets of the invention may further comprise, incorporated into one or more of the masses for processing, a gas-Said gas-evolving effervescent system. evolving effervescent system may consist of a single substance which on contact with water releases a gas. Among these in particular, compounds mention may be made, magnesium peroxide, which on contact with water releases oxygen. Normally, however, the gas-releasing effervescent system consists in its turn of at least two constituents which react with one another and, in so doing, form gas. Although a multitude of systems for example, nitrogen, release, hydrogen are conceivable and implementable here, the effervescent system used in the laundry detergent and cleaning product tablets of the invention will be basis of both economic and selectable the on environmental considerations. Preferred effervescent systems consist of alkali metal carbonate and/or alkali metal hydrogen carbonate and of an acidifier apt to release carbon dioxide from the alkali metal salts in aqueous solution.

Among the alkali metal carbonates and/or alkali metal hydrogen carbonates, the sodium and potassium salts are much preferred over the other salts on grounds of cost. It is of course not mandatory to use the single alkali metal carbonates or alkali metal hydrogen carbonates in question; rather, mixtures of different carbonates and hydrogen carbonates may be preferred from the standpoint of wash technology.

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In preferred laundry detergent and cleaning product tablets, the effervescent system used comprises from 2 to 20% by weight, preferably from 3 to 15% by weight, and in particular from 5 to 10% by weight, of an alkali metal carbonate or alkali metal hydrogen carbonate, and from 1 to 15, preferably from 2 to 12, and in particular from 3 to 10, % by weight of an acidifier, based in each case on the total tablet. The amount of said substances in individual masses may very well be higher.

As examples of acidifiers which release carbon dioxide from the alkali metal salts in aqueous solution it is possible to use boric acid and also alkali metal hydrogen sulfates, alkali metal hydrogen phosphates, Preference is inorganic salts. and other however, to the use of organic acidifiers, with citric acid being a particularly preferred acidifier. However, it is also possible, in particular, to use the other solid mono-, oligo- and polycarboxylic acids. Preferred among this group, in turn, are tartaric acid, succinic acid, malonic acid, adipic acid, maleic acid, fumaric oxalic acid, and polyacrylic acid. sulfonic acids such as amidosulfonic acid may likewise be used. A commercially available acidifier which is likewise preferred for use in the context of the

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present invention is Sokalan® DCS (trademark of BASF), a mixture of succinic acid (max. 31% by weight), glutaric acid (max. 50% by weight), and adipic acid (max. 33% by weight).

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In the context of the present invention, preference is given to laundry detergent and cleaning product tablets where the acidifier used in the effervescent system comprises a substance from the group of the organic di-, tri- and oligocarboxylic acids, and mixtures thereof.

In processes which are preferred in the context of the present invention, at least one of the deformable masses comprises bleaches from the group of the oxygen or halogen bleaches, especially the chlorine bleaches, with particular preference to sodium perborate and sodium percarbonate, in amounts of from 2 to 25% by weight, preferably from 5 to 20% by weight, and in particular from 10 to 15% by weight, based in each case on the mass. These substances are described below.

Among the compounds used as bleaches which yield H₂O₂ in water, particular importance is possessed by sodium percarbonate. This "sodium percarbonate" is a term used unspecifically for sodium carbonate peroxohydrates, which strictly speaking are not "percarbonates" (i.e., salts of percarbonic acid) but rather hydrogen peroxide adducts onto sodium carbonate. The commercial product has the average composition 2 Na₂CO₃·3 H₂O₂ and is thus not a peroxycarbonate. Sodium percarbonate forms a white, water soluble powder of density 2.14 g cm⁻³ which breaks down readily into sodium carbonate and oxygen having a bleaching or oxidizing action.

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Sodium carbonate peroxohydrate was first obtained in 1899 by precipitation with ethanol from a solution of sodium carbonate in hydrogen peroxide, but was

mistakenly regarded as a peroxycarbonate. Only in 1909 was the compound recognized as the hydrogen peroxide addition compound; nevertheless, the historical name (sodium percarbonate) has persisted in the art.

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percarbonate is produced sodium Industrially, predominantly by precipitation from aqueous solution (known as the wet process). In this process, aqueous solutions of sodium carbonate and hydrogen peroxide are combined and the sodium percarbonate is precipitated by (predominantly agents salting οf means example polycrystallizing aids (for chloride). and stabilizers (for phosphates, polyacrylates), example, Mg2+ ions). The precipitated salt, which still contains from 5 to 12% by weight of the mother liquor, is subsequently centrifuged and dried in fluidized-bed driers at 90°C. The bulk density of the finished product may vary between 800 and 1200 g/l according to the production process. Generally, the percarbonate is stabilized by an additional coating. Coating processes, substances used for the coating, are described in the patent literature. Fundamentally, it is possible in accordance with the invention to use all commercially customary percarbonate types, as supplied, for example, by the companies Solvay Interox, Degussa, Kemira or Akzo.

Further bleaches which may be used are, for example, sodium perborate tetrahydrate and sodium perborate monohydrate, peroxypyrophosphates, citrate perhydrates, and $\rm H_2O_2$ -donating peracidic salts or peracids, such as perbenzoates, peroxophthalates, diperazelaic acid, phthaloimino peracid or diperdodecanedioic acid. With the use of the bleaches, as well, it is possible to refrain from the use of surfactants and/or builders, thereby making it possible to produce straight bleach tablets. If such tablets are to be used for bleaching textile laundry, preference is given to a combination

of sodium percarbonate with sodium sesquicarbonate, irrespective of what other ingredients are present in the tablets. If cleaning product tablets or bleach tablets for machine dishwashing are being produced, then the bleaches used may also comprise bleaches from the group of organic bleaches. Typical organic bleaches are the diacyl peroxides, such as dibenzoyl peroxide, for example. Further typical organic bleaches are the peroxy acids, particular examples being the alkyl peroxy acids and the aryl peroxy acids. Preferred representatives are (a) peroxybenzoic acid and ring-substituted derivatives, such as alkylperoxybenzoic acids, and also $peroxy-\alpha-naphthoic$ acid and monoperphthalate, (b) aliphatic or magnesium acids, such as peroxy substituted aliphatic peroxylauric acid, peroxystearic acid, ε-phthalimidoperoxy caproic acid [phthaloiminoperoxyhexanoic acid o-carboxybenzamidoperoxycaproic (PAP)], and N-nonenylamidoacid nonenylamidoperadipic persuccinates, and (c) aliphatic and araliphatic peroxy 1,12-diperoxydecanesuch as acids, dicarboxylic dicarboxylic acid, 1,9-diperoxyazelaic acid, diperoxysebacic acid, diperoxybrassylic acid, the diperoxyphthalic acids, 2-decyldiperoxybutane-1,4-dioic acid and N,N-terephthaloyldi(6-aminopercaproic acid) may be used.

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Bleaches in tablets for machine dishwashing may also be substances which release chlorine or bromine. Among or bromine-releasing materials, suitable chlorineexamples include heterocyclic N-bromoamides and Nchloroamides, examples being trichloroisocyanuric acid, dibromoisocyanuric tribromoisocyanuric acid, and/or dichloroisocyanuric acid (DICA) and/or salts thereof with cations such as potassium and sodium. such as 1,3-dichloro-5,5compounds, Hydantoin dimethylhydantoin, are likewise suitable.

In processes which are further preferred in accordance with the invention, at least one of the deformable masses comprises bleach activators from the groups of polyacylated alkylenediamines, especially tetraacetylethylenediamine (TAED), N-acyl imides, especially Nnonanoylsuccinimide (NOSI), acylated phenolsulfonates, n-nonanoylespecially isononanoyloxybenzenesulfonate (n- or iso-NOBS) and Nmethylmorpholiniumacetonitrile methyl sulfate (MMA), in amounts of from 0.25 to 15% by weight, preferably from 0.5 to 10% by weight, and in particular from 1 to 5% by based in each case on the mass. These substances too are described below.

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In order to achieve an improved bleaching effect when 15 washing or cleaning at temperatures of 60°C and below, it is possible to incorporate bleach activators. Bleach activators, which boost the action of the bleaches, are for example, compounds containing one or more N-acyl and/or O-acyl groups, such as substances from the class 20 acylated anhydrides, esters, and imides the Examples tetraacetylimidazoles oximes. are or tetraacetylmethylene-diamine (TAED), ethylenediamine (TAMD), and tetraacetylhexylenediamine (TAHD), and also 1,5-diacetyl-2,2-(PAG), pentaacetylglucose 25 (DADHT), isatoic and dioxohexahydro-1,3,5-triazine anhydride (ISA).

Bleach activators which may be used are compounds which under perhydrolysis conditions give rise to aliphatic peroxo carboxylic acids having preferably 1 to 10 carbon atoms, in particular 2 to 4 carbon atoms, and/or substituted or unsubstituted perbenzoic acid. Suitable substances are those which carry O-acyl and/or N-acyl groups of the stated number of carbon atoms, and/or substituted or unsubstituted benzoyl groups. Preference is given to polyacylated alkylenediamines, especially tetraacetylethylenediamine (TAED), acylated triazine

1,5-diacetyl-2,4-dioxohexaespecially derivatives, (DADHT), acylated glycolurils, hydro-1,3,5-triazine especially tetraacetylglycoluril (TAGU), N-acyl imides, especially N-nonanoylsuccinimide (NOSI), acylated especially n-nonanoylorphenolsulfonates, (niso-NOBS), nonanoyloxybenzenesulfonate orcarboxylic anhydrides, especially phthalic anhydride, acylated polyhydric alcohols, especially triacetin, ethylene glycol diacetate, 2,5-diacetoxy-2,5-dihydrofuran, N-methylmorpholiniumacetonitrile methyl sulfate (MMA), and the enol esters known from German Patent Applications DE 196 16 693 and DE 196 16 767, and also acetylated sorbitol and mannitol and/or mixtures derivatives, sugar acylated (SORMAN), thereof (PAG), pentaacetylpentaacetylglucose especially 15 fructose, tetraacetylxylose and octaacetyllactose, optionally N-alkylated glucamine acetylated, gluconolactone, and/or N-acylated lactams, for example, Hydrophilically substituted N-benzoylcaprolactam. acylacetals and acyllactams are likewise used with 20 of conventional bleach Combinations preference. activators may also be used.

In addition to the conventional bleach activators, or instead of them, it is also possible to incorporate what are known as bleaching catalysts into the rinse aid particles. These substances are bleach-boosting transition metal salts or transition metal complexes such as, for example, Mn-, Fe-, Co-, Ru- or Mo-salen complexes or -carbonyl complexes. Other bleaching catalysts which can be used include Mn, Fe, Co, Ru, Mo, Ti, V and Cu complexes with N-containing tripod and also Co-, Fe-, Cuand Ru-ammine ligands, complexes.

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Preference is given to the use of bleach activators of polyacylated alkylenediamines, from the group especially tetraacetylethylenediamine (TAED),

imides, especially N-nonanoylsuccinimide (NOSI), acylated phenolsulfonates, especially n-nonanoyl- or isononanoyloxybenzenesulfonate (n- or iso-NOBS), N-methylmorpholiniumacetonitrile methyl sulfate (MMA), preferably in amounts of up to 10% by weight, in particular from 0.1% by weight to 8% by weight, more particularly from 2 to 8% by weight, and with particular preference from 2 to 6% by weight, based on the overall composition.

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Bleach-boosting transition metal complexes, especially those with the central atoms Mn, Fe, Co, Cu, Mo, V, Ti and/or Ru, preferably selected from the group of manganese and/or cobalt salts and/or complexes, with particular preference from cobalt ammine complexes, cobalt acetato complexes, cobalt carbonyl complexes, the chlorides of cobalt or manganese, and manganese sulfate, are used in customary amounts, preferably in an amount of up to 5% by weight, in particular from 0.0025% by weight to 1% by weight, and with particular preference from 0.01% by weight to 0.25% by weight, based in each case on the overall composition. In specific cases, however, it is also possible to use a greater amount of bleach activator.

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Owing to their oxidizing action it is advantageous to separate the bleaches from other ingredients, this purpose being suitably met in particular by processes of the invention for producing multiphase tablets. wherein one of the deformable masses Processes deformable mass while another comprises bleaches comprises bleach activators are preferred.

A further preferred process is one wherein at least one of the deformable masses comprises silver protectants from the group of the triazoles, benzotriazoles, bisbenzotriazoles, aminotriazoles, alkylaminotriazoles and the transition metal salts or transition metal

complexes, with particular preference benzotriazole and/or alkylaminotriazole, in amounts of from 0.01 to 5% by weight, preferably from 0.05 to 4% by weight, and in particular from 0.5 to 3% by weight, based in each case on the mass.

Said corrosion inhibitors may likewise be incorporated into the masses for processing in order to protect the ware or the machine, with special importance in the field of machine dishwashing being possessed, 10 particular, by silver protectants. The known substances of the prior art may be used. In general it is possible to use, in particular, silver protectants selected from the group consisting of triazoles, benzotriazoles, bisbenzotriazoles, aminotriazoles, alkylaminotriazoles, 15 salts or transition transition metal complexes. Particular preference is given to the use of benzotriazole and/or alkylaminotriazole. Frequently encountered in cleaning formulations, furthermore, are chlorine, active containing agents 20 significantly reduce corrosion of the silver surface. In chlorine-free cleaners, use is made in particular of and nitrogen-containing oxygen-containing redox-active compounds, such as divalent and trivalent pyrocatechol, hydroquinone, phenols, e.q. 25 phloroglucinol, gallic acid, hydroxyhydroquinone, and derivatives of classes of these compound. Inorganic compounds in the form of salts and complexes, such as salts of the metals Mn, Ti, Zr, Hf, application. also find frequent and Ce, 30 Preference is given in this context to the transition metal salts selected from the group consisting of manganese and/or cobalt salts and/or complexes, with particular preference cobalt ammine complexes, cobalt carbonyl complexes, acetato complexes, cobalt 35 chlorides of cobalt or of manganese and manganese sulfate. Similarly, zinc compounds may be used to prevent corrosion on the ware.

If corrosion inhibitors are used in multiphase tablets, it is preferred to separate them from the bleaches. Accordingly, processes wherein one of the deformable masses comprises bleaches while another deformable mass comprises corrosion inhibitors are preferred.

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The separation of the bleaches from other ingredients may also be advantageous. Process of the invention wherein one of the deformable masses comprises bleaches while another deformable mass comprises enzymes are include preferred. Suitable enzymes likewise particular those from the classes of the hydrolases such as the proteases, esterases, lipases or lipolytic cellulases or other glycosyl amylases, enzymes, and mixtures of said enzymes. hydrolases, laundry, all of these hydrolases contribute to removing stains, such as proteinaceous, fatty or starchy marks and graying. Cellulases and other glycosyl hydrolases may, furthermore, contribute, by removing pilling and microfibrils, to the retention of color and to an increase in the softness of the textile. For bleaching, inhibiting color transfer it and/or for possible to use oxidoreductases. Especially suitable enzymatic active substances are those obtained from 25 bacterial strains or fungi such as Bacillus subtilis, Bacillus licheniformis, Streptomyces griseus, Coprinus Humicola insolens, and also and cinereus genetically modified variants thereof. Preference is given to the use of proteases of the subtilisin type, 30 and especially to proteases obtained from Bacillus lentus. Of particular interest in this context are enzyme mixtures, examples being those of protease and amylase or protease and lipase or lipolytic enzymes, or protease and cellulase or of cellulase and lipase or lipolytic enzymes of protease, amylase and lipase or lipolytic enzymes, or protease, lipase or lipolytic enzymes and cellulase, but especially protease and/or

lipase-containing mixtures or mixtures with lipolytic enzymes. Examples of such lipolytic enzymes are the known cutinases. Peroxidases or oxidases have also proven suitable in some cases. The suitable amylases include, in particular, alpha-amylases, iso-amylases, Cellulases used and pectinases. pullulanases, cellobiohydrolases, endoglucanases and preferably endoglucosidases, which are also called cellobiases, mixtures thereof. Because different types and their CMCase and Avicelase differ in cellulase activities, specific mixtures of the cellulases may be used to establish the desired activities.

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In cleaning product tablets for machine dishwashing, naturally, different enzymes are used in order to take 15 treated the different substrates account of different types of soiling. Suitable enzymes here include in particular those from the classes of the hydrolases such as the proteases, esterases, lipases or lipolytic enzymes, amylases, glycosyl hydrolases, and 20 mixtures of said enzymes. All of these hydrolases contribute to removing stains, such as proteinaceous, fatty or starchy marks. For bleaching, it is also possible to use oxidoreductases. Especially suitable enzymatic active substances are those obtained from 25 bacterial strains or fungi such as Bacillus subtilis, Bacillus licheniformis, Streptomyces griseus, Coprinus also Humicola insolens, and and cinereus genetically modified variants thereof. Preference is given to the use of proteases of the subtilisin type, 30 and especially to proteases obtained from Bacillus lentus. Of particular interest in this context are enzyme mixtures, examples being those of protease and amylase or protease and lipase or lipolytic enzymes, or of protease, amylase and lipase or lipolytic enzymes, 35 lipolytic enzymes, lipase or protease, especially protease and/or lipase-containing mixtures or mixtures with lipolytic enzymes. Examples of such lipolytic enzymes are the known cutinases. Peroxidases or oxidases have also proven suitable in some cases. The suitable amylases include, in particular, alphaamylases, iso-amylases, pullulanases, and pectinases.

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The enzymes may be adsorbed on carrier substances or embedded in sheathing substances in order to protect them against premature decomposition. The proportion of the enzymes, enzyme mixtures or enzyme granules may be, for example, from about 0.1 to 5% by weight, preferably from 0.5 to about 4.5% by weight, based in each case on the mass(es).

Irrespective of the intended application of the tablets produced in accordance with the invention (for example, laundry detergent tablets or cleaning product tablets), preference is given to processes wherein one of the material strands emerging from the emergence apertures comprises enzymes.

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Enzyme-containing masses of this kind are preferably processed in multistrand processes; i.e., in addition to a material strand comprising enzymes, there exists at least one further strand which is preferably free from enzymes. Here, particular preference is given to processes wherein the enzyme-containing material strand is enveloped by an enzyme-free material.

from the bleaches Separation the of described earlier on above may also be advantageous, so 30 that preferred processes are those wherein one of the deformable masses comprises bleaches while another surfactants, preferably deformable comprises mass preference with particular nonionic surfactants, alkoxylated alcohols having 10 to 24 carbon atoms and 35 from 1 to 5 alkylene oxide units.

Further ingredients which may, in the context of the process of the invention, be part of one or more masses are, for example, cobuilders (see above), dyes, optical brighteners, fragrances, soil release compounds, soil repellents, antioxidants, fluorescence agents, inhibitors, silicone fluids and/or liquid paraffins, inhibitors. inhibitors, graying transfer substances are These boosters, etc. detergency described below.

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Organic builder substances which may be used are, for example, the polycarboxylic acids, usable in the form of their sodium salts, the term polycarboxylic acids meaning those carboxylic acids which carry more than one acid function. Examples of these are citric acid, adipic acid, succinic acid, glutaric acid, malic acid, tartaric acid, maleic acid, fumaric acid, sugar acids, amino carboxylic acids, nitrilotriacetic acid (NTA), provided such use is not objectionable on ecological grounds, and also mixtures thereof. Preferred salts are the salts of the polycarboxylic acids such as citric acid, adipic acid, succinic acid, glutaric acid, tartaric acid, sugar acids, and mixtures thereof.

The acids per se may also be used. In addition to their builder effect, the acids typically also possess the property of an acidifying component and thus also serve to establish a lower and milder pH of laundry detergents or cleaning products. In this context, mention may be made in particular of citric acid, succinic acid, glutaric acid, adipic acid, gluconic acid, and any desired mixtures thereof.

Also suitable as builders are polymeric polycarboxylates; these are, for example, the alkali metal salts of polyacrylic acid or of polymethacrylic acid, examples being those having a relative molecular mass of from 500 to 70,000 g/mol. The molecular masses reported for polymeric polycarboxylates, for the purposes of this document, are weight-average molecular masses, M_{w} , of the respective determined basically by means acid form, permeation chromatography (GPC) using a UV detector. made against an external measurement was which owing to its standard, polyacrylic acid the polymers under similarity to structural realistic molecular weight provides investigation markedly from figures differ These values. using polyobtained values weight molecular styrenesulfonic acids as the standard. The molecular masses measured against polystyrenesulfonic acids are masses than the molecular generally much higher reported in this document.

Suitable polymers are, in particular, polyacrylates, which preferably have a molecular mass of from 2000 to 20,000 g/mol. Owing to their superior solubility, preference in this group may be given in turn to the short-chain polyacrylates, which have molecular masses of from 2000 to 10,000 g/mol, and with particular preference from 3000 to 5000 g/mol.

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polycarboxylates, copolymeric suitable are Also especially those of acrylic acid with methacrylic acid and of acrylic acid or methacrylic acid with maleic acid. Copolymers which have been found particularly suitable are those of acrylic acid with maleic acid which contain from 50 to 90% by weight acrylic acid and from 50 to 10% by weight maleic acid. Their relative molecular mass, based on free acids, is generally from 2000 to 70,000 g/mol, preferably from 20,000 to 30,000 to and in particular from 50,000 g/mol, 40,000 q/mol.

The (co)polymeric polycarboxylates can be used either as powders or as aqueous solutions. The (co)polymeric polycarboxylate content of the compositions is preferably from 0.5 to 20% by weight, in particular from 3 to 10% by weight.

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In order to improve the solubility in water, the polymers may also contain allylsulfonic acids, such as allyloxybenzenesulfonic acid and methallylsulfonic acid, for example, as monomers.

Particular preference is also given to biodegradable polymers comprising more than two different monomer units, examples being those comprising, as monomers, salts of acrylic acid and of maleic acid, and also vinyl alcohol or vinyl alcohol derivatives, or those comprising, as monomers, salts of acrylic acid and of 2-alkylallylsulfonic acid, and also sugar derivatives.

Further preferred copolymers are those described in German Patent Applications DE-A-43 03 320 and DE-A-44 17 734, whose monomers are preferably acrolein and acrylic acid/acrylic acid salts, and, respectively, acrolein and vinyl acetate.

Similarly, further preferred builder substances that may be mentioned include polymeric amino dicarboxylic acids, their salts or their precursor substances. Particular preference is given to polyaspartic acids and their salts and derivatives, which are disclosed in German Patent Application DE-A-195 40 086 to have not only cobuilder properties but also a bleach-stabilizing action.

Further suitable builder substances are polyacetals, which may be obtained by reacting dialdehydes with polyol carboxylic acids having 5 to 7 carbon atoms and at least 3 hydroxyl groups. Preferred polyacetals are

obtained from dialdehydes such as glyoxal, glutaraldehyde, terephthalaldehyde and mixtures thereof and from polyol carboxylic acids such as gluconic acid and/or glucoheptonic acid.

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builder substances are organic suitable Further dextrins, examples being oligomers and polymers of obtained carbohydrates, which may be by hydrolysis of starches. The hydrolysis can be conducted by customary processes; for example, acid-catalyzed or enzyme-catalyzed processes. The hydrolysis products preferably have average molecular masses in the range from 400 to 500,000 g/mol. Preference is given here to a polysaccharide having a dextrose equivalent (DE) in the range from 0.5 to 40, in particular from 2 to 30, DE being a common measure of the reducing effect of a to dextrose, comparison polysaccharide in possesses a DE of 100. It is possible to use both maltodextrins having a DE of between 3 and 20 and dried glucose syrups having a DE of between 20 and 37, and also so-called yellow dextrins and white dextrins having higher molecular masses, in the range from 2000 to 30,000 g/mol.

The oxidized derivatives of such dextrins comprise 25 their products of reaction with oxidizing agents which are able to oxidize at least one alcohol function of the saccharide ring to the carboxylic acid function. Oxidized dextrins of this kind, and processes for preparing them, are known, for example, from European 30 Patent Applications EP-A-0 232 202, EP-A-0 427 349, 542 496 and and EP-A-0 EP-A-0 472 042 International Patent Applications WO 92/18542, 93/08251, WO 93/16110, WO 94/28030, WO 95/07303, 95/12619 and WO 95/20608. Likewise suitable is 35 oxidized oligosaccharide in accordance with German Patent Application DE-A-196 00 018. A product oxidized at C_6 of the saccharide ring may be particularly advantageous.

Oxydisuccinates and other derivatives of disuccinates, preferably ethylenediamine disuccinate, are suitable cobuilders. Ethylenediamine N,N'-disuccinate (EDDS) is used preferably in the form of its sodium or magnesium salts. Further preference in this context is and disuccinates glycerol given to amounts use well. Suitable as trisuccinates formulations containing zeolite and/or silicate are from 3 to 15% by weight.

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Examples of further useful organic cobuilders are acetylated hydroxy carboxylic acids and their salts, which may also be present in lactone form and which contain at least 4 carbon atoms, at least one hydroxyl group, and not more than two acid groups. Such cobuilders are described, for example, in International Patent Application WO 95/20029.

cobuilder having class of substance further properties is represented by the phosphonates. particular, in are, phosphonates in question hydroxyalkane- and aminoalkanephosphonates. Among the 1-hydroxyethane-1,1-diphoshydroxyalkanephosphonates, is of particular importance phonate (HEDP) cobuilder. It is used preferably as the sodium salt, the disodium salt being neutral and the tetrasodium salt giving an alkaline (pH 9) reaction. aminoalkanephosphonates are preferably ethylenediaminetetramethylenephosphonate (EDTMP), diethylenetriaminepentamethylenephosphonate (DTPMP), and their higher homologs. They are used preferably in the form of the salts, e.g., sodium neutrally reacting hexasodium salt of EDTMP or as the hepta- and octasodium salt of DTPMP. As a builder in this case, preference is given to using HEDP from the class of the phosphonates. Furthermore, the aminoalkanephosphonates possess a pronounced heavy metal binding capacity. Accordingly, and especially if the compositions also contain bleach, it may be preferred to use aminoalkanephosphonates, especially DTPMP, or to use mixtures of said phosphonates.

Furthermore, all compounds capable of forming complexes with alkaline earth metal ions may be used as cobuilders.

In order to enhance the esthetic appeal of the detergent tablets of the invention, they may in whole or in part be colored with appropriate dyes. Particular optical effects may be achieved if, where tablets are produced from two or more masses, the masses for processing are differently colored. Preferred dyes, whose selection presents no difficulty whatsoever to the skilled worker, possess a high level of storage stability and insensitivity to the other ingredients of the compositions and to light and possess no pronounced affinity for the substrates treated, such as textile fibers or parts of kitchen- or tableware, so as not to stain them.

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Preference for use in the laundry detergent tablets of the invention is given to all colorants which can be oxidatively destroyed in the wash process, and to mixtures thereof with suitable blue dyes, known as bluing agents. It has proven advantageous to use colorants which are soluble in water or at room temperature in liquid organic substances. Examples of suitable colorants are anionic colorants, e.g., anionic nitroso dyes. One possible colorant is, for example, naphthol green (Colour Index (CI) Part 1: Acid Green 1; Part 2: 10020) which as a commercial product is obtainable, for example, as Basacid® Green 970 from BASF, Ludwigshafen, and also mixtures thereof with

suitable blue dyes. Further suitable colorants include Pigmosol® Blue 6900 (CI 74160), Pigmosol® Green 8730 (CI 74260), Basonyl® Red 545 FL (CI 45170), Sandolan® Rhodamin EB400 (CI 45100), Basacid® Yellow 094 (CI 47005), Sicovit® Patent Blue 85 E 131 (CI 42051), Acid Blue 183 (CAS 12217-22-0, CI Acid Blue 183), Pigment Blue 15 (CI 74160), Supranol® Blue GLW (CAS 12219-32-8, CI Acid Blue 221), Nylosan® Yellow N-7GL SGR (CAS 61814-57-1, CI Acid Yellow 218) and/or Sandolan® Blue (CI Acid Blue 182, CAS 12219-26-0).

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In the context of the choice of colorant it must be ensured that the colorants do not have too great an affinity for the textile surfaces, and especially for synthetic fibers. At the same time, it should also be borne in mind in choosing appropriate colorants that colorants possess different stabilities with respect to oxidation. The general rule is that water-insoluble colorants are more stable to oxidation than watersoluble colorants. Depending on the solubility oxidation sensitivity, the also on concentration of the colorant in the laundry detergents and cleaning products varies. With readily watersoluble colorants, e.g., the abovementioned Basacid® Green, or the likewise abovementioned Sandolan® Blue, color concentrations chosen are typically in the range from a few 10^{-2} to 10^{-3} % by weight. In the case of the pigment dyes, which are particularly preferred for reason of their brightness but are less readily soluble in water, examples being the abovementioned Pigmosol® dyes, the appropriate concentration of the colorant in laundry detergents or cleaning products, in contrast, is typically from a few 10^{-3} to 10^{-4} % by weight.

35 The laundry detergent and cleaning product tablets produced by the process of the invention may comprise one or more optical brighteners. These substances, which are also called "whiteners", are used in modern

laundry detergents because even freshly washed and bleached white laundry has a slight yellow cast. Optical brighteners are organic dyes which convert a part of the invisible UV radiation of sunlight into longer-wave blue light. The emission of this blue light fills the "gap" in the light reflected by the textile, so that a textile treated with optical brightener appears whiter and lighter to the eye. Since the mechanism of action of brighteners necessitates that they go onto the fibers, a distinction is made in accordance with the fibers to be "dyed" between, for example, brighteners for cotton, nylon, or polyester fibers. The commercially customary brighteners suitable laundry detergents into incorporation primarily to five structural groups: the stilbene the coumarinthe diphenylstilbene group, quinoline group, the diphenylpyrazoline group, and the benzoxazole combination of involving group benzimidazole with conjugated systems. An overview of current brighteners can be found, for example, in G. Jakobi, A. Löhr, "Detergents and Textile Washing", VCH-Verlag, Weinheim, 1987, pages 94 to 100. Examples of suitable brighteners are salts of 4,4'-bis[(4-anilino-6-morpholino-s-triazin-2-yl)amino|stilbene-2,2'-disulfonic acid or compounds of similar structure which instead of the morphilino group carry a diethanolamino group, a methylamino group, an anilino group, or a 2-methoxyethylamino group. Furthermore, brighteners of the substituted diphenylstyryl type may be present, examples being the alkali metal salts of 4,4'-bis(2-30 sulfostyryl)biphenyl, 4,4'-bis(4-chloro-3-sulfostyryl)-4-(4-chlorostyryl)-4'-(2-sulfostyryl)biphenyl, orbiphenyl. Mixtures of the abovementioned brighteners may also be used.

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added to the compositions Fragrances are invention in order to enhance the esthetic appeal of the products which are formed and to provide the

consumer with not only the performance but also a visually and sensorially "typical and unmistakeable" product. As perfume oils and/or fragrances it possible to use individual odorant compounds, examples being the synthetic products of the ester, ether, ketone, alcohol, and hydrocarbon types. aldehyde, Odorant compounds of the ester type are, for example, benzyl acetate, phenoxyethyl isobutyrate, p-tert-butylacetate, dimethyllinalyl acetate, cvclohexyl benzylcarbinyl acetate, phenylethyl acetate, 10 benzoate, benzyl formate, ethyl methylphenylglycinate, allyl cyclo-hexylpropionate, styrallyl propionate, and benzyl salicylate. The ethers include, for example, benzyl ethyl ether; the aldehydes include, for example, the linear alkanals having 8-18 carbon atoms, citral, 15 citronellyloxyacetaldehyde, citronellal, aldehyde, hydroxycitronellal, lilial and bourgeonal; example, the ionones, include, for ketones the ketone; cedryl and methyl α -isomethylionone citronellol, anethole, alcohols include 20 geraniol, linalool, phenylethyl alcohol, and terpineol; the hydrocarbons include primarily the terpenes such as limonene and pinene. Preference, however, is given to use of mixtures of different odorants, which together produce an appealing fragrance note. 25 perfume oils may also contain natural odorant mixtures, as obtainable from plant sources, examples being pine oil, citrus oil, jasmine oil, patchouli oil, rose oil or ylang-ylang oil. Likewise suitable are clary sage oil, camomile oil, clove oil, balm oil, mint oil, cinnamon leaf oil, lime blossom oil, juniperberry oil, vetiver oil, olibanum oil, galbanum oil and labdanum oil, and also orange blossom oil, neroliol, orange peel oil, and sandalwood oil.

The fragrance content of the laundry detergent and cleaning product tablets prepared in accordance with the invention is usually up to 2% by weight of the

overall formulation. The fragrances may be incorporated directly into the compositions of the invention; alternatively, it may be advantageous to apply the fragrances to carriers which intensify the adhesion of the perfume on the laundry and, by means of slower fragrance release, ensure long-lasting fragrance of the textiles. Materials which have become established as such carriers are, for example, cyclodextrins, it being possible in addition for the cyclodextrin-perfume complexes to be additionally coated with further auxiliaries.

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In addition, the laundry detergent and cleaning product tablets may also comprise components which have a positive influence on the ease with which oil and 15 grease are washed off from textiles (these components being known as soil repellents). This effect becomes particularly marked when a textile is soiled that has already been laundered previously a number of times with a detergent of the invention comprising this oil-20 and fat-dissolving component. The preferred oil- and for example, include, components fat-dissolving nonionic cellulose ethers such as methylcellulose and methylhydroxypropylcellulose having a methoxy group content of from 15 to 30% by weight and a hydroxypropyl 25 group content of from 1 to 15% by weight, based in each case on the nonionic cellulose ether, and also the prior art polymers of phthalic acid and/or terephthalic acid, and/or derivatives thereof, especially polymers of ethylene terephthalates and/or polyethylene glycol 30 or anionically and/or nonionically terephthalates modified derivatives thereof. Of these, preference is given to the sulfonated derivatives of terephthalic phthalic acid polymers and of polymers. 35

Foam inhibitors which may be used in the compositions produced in accordance with the invention are suitably,

for example, soaps, paraffins or silicone oils, which may if desired have been applied to carrier materials.

Graying inhibitors have the function of keeping the dirt detached from the fiber in suspension in the liquor and so preventing the redeposition of the dirt. Suitable for this purpose are water-soluble colloids, usually organic in nature, examples being the watersoluble salts of polymeric carboxylic acids, gelatin, salts of ethersulfonic acids of starch or of salts of acidic sulfuric esters cellulose, or starch. Water-soluble polyamides of cellulose orcontaining acidic groups are also suitable for this purpose. Furthermore, soluble starch preparations and starch products other than those mentioned above may be starch, aldehyde being degraded examples starches, etc. Polyvinylpyrrolidone may also be used. Preference, however, is given to the use of cellulose carboxymethylcellulose (Na such as ethers hydroxyalkylcellulose, mixed and methylcellulose, ethers such as methylhydroxyethylcellulose, methylmethylcarboxymethylcellulose hydroxypropylcellulose, and mixtures thereof in amounts of from 0.1 to 5% by weight, based on the compositions.

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Since sheetlike textile structures, especially those of cotton and blends filament rayon, viscose rayon, thereof, may tend to crease, because the individual bending, buckling, to susceptible fibers are the compressing and pinching transverse to direction, the compositions produced in accordance with the invention may comprise synthetic crease control agents. These include, for example, synthetic products based on fatty acids, fatty acid esters, fatty acid fatty alkylol esters, fatty acid amides, alkylolamides, or fatty alcohols, which are usually reacted with ethylene oxide, or else products based on lecithin or on modified phosphoric esters.

In order to combat microorganisms, the compositions produced in accordance with the invention may comprise antimicrobial active substances. In this context a antimicrobial depending on made, distinction is spectrum and mechanism of action, between bacteriostats fungiostats and fungicides, and bactericides, Examples of important substances from these groups are alkylarylsulfonates, halochlorides, benzalkonium and phenylmercuric acetate, it also being phenols, possible to do without these compounds entirely.

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In order to prevent unwanted changes to the compositions and/or the treated textiles as a result of oxygen exposure and other oxidative processes, the compositions may comprise antioxidants. This class of compound includes, for example, substituted phenols, hydroquinones, pyrocatechols and aromatic amines, and also organic sulfides, polysulfides, dithiocarbamates, phosphites, and phosphonates.

Increased wear comfort may result from the additional use of antistats which are further added to compositions produced in accordance with the invention. Antistats increase the surface conductivity and thus enable better dissipation of charges that are formed. External antistats are generally substances having at least one hydrophilic molecule ligand, and provide a more or less hygroscopic film on the surfaces. These antistats, which are usually surface-active, may be subdivided into nitrogen-containing (amines, amides, quaternary ammonium compounds), phosphorus-containing (alkylsulfur-containing esters), and (phosphoric antistats. External sulfates) alkyl sulfonates, Patent described, example, in antistats are for Applications FR 1,156,513, GB 873 214 and GB 839 407. stearyl-)dimethylbenzylammonium (or lauryl-The chlorides disclosed here are suitable as antistats for textiles and as additives to laundry detergents, in which case, additionally, a hand effect is obtained.

In order to improve the water absorption capacity, the textiles, treated rewettability of the facilitate ironing of the treated textiles, silicone may be used example, derivatives, for compositions produced in accordance with the invention. These derivatives additionally improve the rinse-out behavior of the compositions, by virtue of their foam inhibiting properties. Examples of preferred silicone derivatives are polydialkylsiloxanes or alkylarylsiloxanes where the alkyl groups have one to five carbon atoms and are totally or partially fluorinated. Preferred silicones are polydimethylsiloxanes, which may if desired have been derivatized and in that case are amino-functional or quaternized, or have Si-OH, The viscosities of Si-H and/or Si-Cl bonds. preferred silicones at 25°C are in the range between 100 and 100,000 centistokes, it being possible to use the silicones in amounts of between 0.2 and 5% by weight, based on the overall composition.

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Finally, the compositions produced in accordance with the invention may also comprise UV absorbers, which attach to the treated textiles and improve the light stability of the fibers. Compounds which have these desired properties are, for example, the compounds which are active via radiationless deactivation, and derivatives of benzophenone having substituents in position(s) 2 and/or 4. Also suitable are substituted benzotriazoles, acrylates which are phenyl-substituted in position 3 (cinnamic acid derivatives), with or without cyano groups in position 2, salicylates, organic Ni complexes, and also natural substances such as umbelliferone and the endogenous urocanic acid.

In the course of the above remarks, mention has occasionally been made of the amount of the individual substances in the end products of the process of the invention. Based on the masses to be processed, preference is generally given to processes wherein at least one of the deformable masses further comprises one or more substances from the groups of enzymes, corrosion inhibitors, scale inhibitors, cobuilders, dyes and/or fragrances in total amounts of from 6 to 30% by weight, preferably from 7.5 to 25% by weight, and in particular from 10 to 20% by weight, based in each case on the mass.

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abovementioned ingredients, of the all With advantageous properties may result from separating them 15 from other ingredients and/or from formulating them together with certain other ingredients. In the case of multiphase tablets, the individual phases may also differ in the amount they contain of the ingredient, as a result of which advantages may be 20 wherein at least two of achieved. Processes deformable masses comprise the same active subtonic in different amounts are preferred. The term "different amount" relates in this case, as already explained, not to the absolute amount of the ingredient in the mass 25 but to the relative amount based on the phase weight; in other words, it is a % by weight based on the individual mass.

The end products of the process of the invention may be 30 provided in a very wide variety of geometric forms, this flexibility being one of the many advantages of the process of the invention. It is also possible, however, in accordance with the invention to produce in their appearance tablets which are similar 35 example, they be For conventional tablets. manufactured in predetermined three-dimensional forms and predetermined sizes, suitable three-dimensional

forms being virtually any practicable designs - i.e., for example, bar, rod or ingot form, cubes, blocks, and corresponding three-dimensional elements having planar side faces, and in particular cylindrical designs with a circular or oval cross section. This last design covers forms ranging from tablets through to compact cylinders having a height-to-diameter ratio of more than 1.

The end products of the process of the invention may in 10 each case be formed as separate, individual elements corresponding to the predetermined dosage of laundry detergents and/or cleaning products. It equally possible, however, to design the cut-to-length material strands in such a way as to combine a 15 plurality of such mass units in one compact, with the ease of separation of smaller, portioned units being provided for in particular by means of predetermined For the use of textile breakage points. detergents in machines of the type customary in Europe, 20 with a horizontally arranged mechanism, it may as tablets, design the compacts judicious to cylindrical or block form, preference being given to a diameter/height ratio in the range from about 0.5:2 to 2:0.5. 25

The three-dimensional form of another embodiment of the tablets is adapted in its dimensions to the dispensing cup of commercially customary washing machines, so that the tablets can be metered without a dosing aid directly into the dispensing cup, where they dissolve during the initial rinse cycle. Alternatively, it is of course readily possible, and preferred in the context of the present invention, to use the laundry detergent tablets by way of a dosing aid.

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A further preferred tablet which may be produced as a platelike or barlike structure with, in alternation,

segments, short, thin thick and long, individual segments can be broken off from this "slab" at the predetermined breakage points, represented by segments, and inserted thin short, "slablike" laundry machine. This principle of the in other be realized detergent tablet may also example, vertical triangles geometric forms, for connected to one another lengthwise at only one of their sides.

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"Slablike" strand sections of this kind may be produced, after cutting to length, by means of an aftertreatment step which comprises pressing a second blade or a second set of blades into the cut-to-length strand sections without dividing them. Superficial shaping or the production of positive or negative indicia may also take place in accordance with the invention. Preferred processes, accordingly, are those wherein the cut-to-length tablets are subjected to an aftertreatment step.

indicia, impression of addition to the In aftertreatment step may also comprise the impression of patterns, shapes, etc. In this way it is possible, for example, to label universal laundry detergents produced in accordance with the invention by a t-shirt symbol, color laundry detergents produced in accordance with the invention by a wool symbol, cleaning product tablets for machine dishwashing produced in accordance with the invention by symbols such as glasses, plates, pots, pans, etc. No limits are imposed in this case on the creativity of product managers. Preferred processes of the invention therefore comprise as aftertreatment step an additional shaping step, especially one of impression.

Subsequent coating of the cut-to-length tablets is also possible, provided the application of an additional

coating is desirable. In this case, then, processes are preferred wherein the aftertreatment step comprises the coating of the tablets with a pourable material, preferably a pourable material having a viscosity < 5000 mPas.

Independently of the number of the phases and the nature of the aftertreatment, preference is generally given to processes wherein the tablets have a density of more than 800 kg dm⁻³, preferably more than 900 kg dm⁻³, with particular preference more than 1000 kg dm⁻³, and in particular more than 1100 kg dm⁻³. In such tablets, the advantages of the commercial form of a compact laundry detergent or cleaning product are manifested particularly clearly.

The present invention provides a process which makes it possible to produce laundry detergent and cleaning product tablets simply and under changing framework conditions. A preferred hardening mechanism in this context, as described above, consists in time-delayed water binding, corresponding laundry detergent and cleaning product tablets having not been described in therefore invention present The art. additionally provides a laundry detergent or cleaning product tablet comprising at least 30% by weight of phosphate(s), wherein the water content of the tablet is from 50 to 100% of the calculated water binding capacity.

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With regard to the definition of the water binding capacity and its calculation, reference may be made to the above remarks, in order to avoid redundancy. The phosphate content of preferred tablets produced in accordance with the invention is higher, so that preferred laundry detergent and cleaning product tablets are those which comprise at least 40% by weight, preferably at least 45% by weight, and in

particular at least 50% by weight, of phosphate(s), based in each case on the tablet weight.

As already mentioned in connection with the remarks 5 relating to the process of the invention, phosphates whose use is particularly preferred are alkali metal analogously, completely Therefore, phosphates. preference is given to laundry detergent and cleaning alkali comprise which tablets product phosphate(s), with particular preference pentasodium 10 pentapotassium triphosphate (sodium potassium tripolyphosphate), in amounts of from 30 to 80% by weight, preferably from 35 to 75% by weight, and in particular from 50 to 70% by weight, based in each case on the tablet weight. 15

In the description of the process of the invention it was explained that process end products which are particularly preferred in the context of the present invention not only possess an extremely low proportion of free water but are preferably themselves still able to bind further free water. In preferred laundry detergent and cleaning product tablets, therefore, the water content of the tablets is from 55 to 95%, preferably from 60 to 90%, and in particular from 65 to 85%, of the calculated water binding capacity.

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With regard to further ingredients, their amounts and physical properties, it is possible to refer to the above remarks, as it is with regard to the multiphase nature of tablets of the invention, the division of ingredients between the individual phases, and the proportions of the phases with respect to one another.

While the invention has been described with particular reference to certain embodiments thereof, it will be understood that changes and modifications may be made

by those of ordinary skill in the art within the scope and spirit of the following claims.

In the claims, the word "comprising" means "including the following elements (in the body), but not excluding others"; the phrase "consisting of" means "excluding more than traces of other than the recited ingredients"; and the phrase "consisting essentially of" means "excluding unspecified ingredients which materially affect the basic characteristics of the composition".

What is claimed is:

- A process for producing laundry detergent and cleaning product tablets, which comprises preparing (a) deformable mass(es), supplying said mass(es) with a pressure below 40 bar to emergence apertures, cutting the emerging material strands to tablet dimensions, and hardening them.
- 10 2. The process as claimed in claim 1, wherein the deformable mass(es) is (are) supplied to the emergence apertures with a pressure below 35 bar.
- 3. The process as claimed in claim 2 wherein the pressure is below 30 bar.
 - 4. The process as claimed in claim 2 wherein the pressure is below 20 bar.
- 20 5. The process as claimed in claim 2 wherein the pressure is below 10 bar.
- 6. The process as claimed in any of claims 1 to 5, wherein the deformable mass(es) is (are) supplied to the emergence apertures with a pressure below 8.5 bar.
 - 7. The process as claimed in claim 6 wherein the pressure is below 7.5 bar.
 - 8. The process as claimed in claim 6 wherein the pressure is below 6.5 bar.

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The process as claimed in claim 6 wherein the
 pressure is below 5 bar.

10. The process as claimed in any of claims 1 to 9, wherein a deformable mass is drawn in between two rolls, discharged as a material strand from emergence apertures, cut to the desired tablet dimension, and hardened.

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- 11. The process as claimed in any of claims 1 to 10, wherein two deformable masses of different composition are drawn in between two roll pairs and discharged as filled, hollow or multi-ply material strands from emergence apertures, cut to the desired tablet dimension, and hardened.
- 15 12. The process as claimed in any of claims 1 to 11, wherein three plastically deformable masses of different composition are drawn in between three roll pairs and discharged as singly, doubly or triply filled, hollow, two- or three-ply material strands from emergence apertures, cut to the desired tablet dimension, and hardened.
- 13. The process as claimed in any of claims 1 to 12, wherein the material strands are discharged from the emergence apertures at a rate of from 0.2 m/min to 30 m/min.
 - 14. The process as claimed in claim 13 wherein the rate is from 0.25 m/min to 20 m/min.
 - 15. The process as claimed in claim 13 wherein the rate is from 0.5 m/min to 15 m/min.
- 16. The process as claimed in claim 13 wherein the rate is from 1 m/min to 10 m/min.

- 17. The process as claimed in any of claims 1 to 16, wherein the emergence apertures have aperture areas of from 50 mm² to 2500 mm².
- 5 18. The process as claimed in claim 17 wherein the areas are from 100 \mbox{mm}^2 to 2000 $\mbox{m}^2.$
 - 19. The process as claimed in claim 17 wherein the areas are from 200 $\,\mathrm{mm}^2$ to 1500 $\,\mathrm{mm}^2$.
- 20. The process as claimed in claim 17 wherein the areas are from 300 mm² to 1000 mm².

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- 21. The process as claimed in claim 17 wherein the areas are from 350 mm^2 to 750 mm^2 .
 - 22. The process as claimed in any of claims 1 to 21, wherein the thickness of at least one of the material strands emerging from the emergence apertures is at least 5 mm.
 - 23. The process as claimed in claim 22 wherein the thickness is at least 7.5 mm.
- 25 24. The process as claimed in claim 22 wherein the thickness is at least 10 mm.
- 25. The process as claimed in any of claims 1 to 24, wherein the material strands emerging from the emergence apertures are cut to a length of from 10 to 100 mm.
 - 26. The process as claimed in claim 25 wherein the length is from 12.5 to 75 mm.
 - 27. The process as claimed in claim 25 wherein the length is from 15 to 60 mm.

- 28. The process as claimed in claim 25 wherein the length is from 20 to 50 mm.
- 29. The process as claimed in any of claims 1 to 28, wherein the hardening of the material strands, cut to tablet dimensions, is assisted by superficial drying and/or cooling, in particular by blowing with cold air.
- 10 30. The process as claimed in claim 29 wherein the drying and/or cooling is assisted by blowing with cold air.
- 31. The process as claimed in any of claims 1 to 30, wherein the deformable mass(es) comprise(s) from 10 to 95% by weight of anhydrous substances which pass by hydration into a hydrate form having a melting point below 120°C.
- 20 32. The process as claimed in claim 31 wherein the deformable masses comprise 15 to 90% by weight.
 - 33. The process as claimed in claim 31 wherein the deformable masses comprise 20 to 85% by weight.
 - 34. The process as claimed in claim 31 wherein the deformable masses comprise 25 to 80% by weight.

- 35. The process of claims 31 to 34, wherein the melting point is below 100°C.
 - 36. The process of claims 31 to 34, wherein the melting point is below 80°C.
- 35 37. The process as claimed in any of claims 1 to 36, wherein the deformable mass(es) comprise(s) phosphate(s) in amounts of from 20 to 80% by weight, based on the mass.

- 38. The process as claimed in claim 37 wherein the phosphate is alkali metal phosphate.
- 39. The process as claimed in claim 37 wherein the phosphate is pentapotassium and/or pentapotassium triphosphate.
- 40. The process as claimed in claim 37 wherein the phosphate is sodium and/or potassium tripolyphosphate.
 - 41. The process as claimed in claims 37 to 40 wherein the amounts are from 25 to 75% by weight.
- 15 42. Theprocess as claimed in claims 37 to 40 wherein the amounts are from 30 to 70% by weight.
- 43. The process as claimed in any of claims 37 to 42, wherein the weight ratio of phosphate(s) to water in the deformable mass is less than 1:0.3.
 - 44. The process as claimed in claim 43 wherein the ratio is less than 1:0.25.
- 25 45. The process as claimed in claim 43 wherein the ratio is less than 1:0.2.
- 46. The process as claimed in any of claims 1 to 45, wherein the deformable mass(es) comprise(s) 30 carbonate(s) and/or hydrogen carbonate(s) in amounts of from 5 to 50% by weight.

- 47. The process as claimed in claim 46 wherein alkali metal carbonate is present.
- 48. The process as claimed in claim 46 wherein sodium carbonate is present.

- 49. The process as claimed in claim 46 to 48 wherein the amount is from 7.5 to 40% by weight.
- 50. The process as claimed in claim 46 to 48 wherein the amount is from 10 to 30% by weight.
 - 51. The process as claimed in any of claims 1 to 50, wherein the deformable mass(es) comprise(s) silicate(s) in amounts of from 10 to 60% by weight based on the mass.

- 52. The process as claimed in claim 51, wherein the silicate(s) is an alkali metal silicate.
- 15 53. The process as claimed in claim 52, wherein the alkali metal silicate is a crystalline or amorphous alkali metal disilicate.
- 54. The process as claimed in any one of claims 51 to 20 53, wherein the amounts are from 15 to 50% by weight.
- 55. The process as claimed in any one of claims 51 to 50, wherein the amounts are from 20 to 40% by weight.
- 56. The process as claimed in any of claims 1 to 55, wherein the deformable mass(es) comprise(s) zeolite(s), preferably zeolite A, zeolite P, zeolite X and mixtures thereof, in amounts of from 10 to 60% by weight, based on the mass.
 - 57. The process as claimed in claim 56, wherein the amount is from 15 to 50% by weight.
 - 58. The process as claimed in claim 56, wherein the amount is from 20 to 40% by weight.

- 59. The process as claimed in any of claims 1 to 58, wherein the average particle size of the solids used in the deformable mass(es) is below 400 μm .
- 60. The process as claimed in claim 59, wherein the size is below 300 $\mu m\,.$

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- 61. The process as claimed in claim 59, wherein the size is below 200 μm .
 - 62. The process as claimed in any of claims 1 to 61, wherein less than 10% by weight of the solids used in the deformable mass(es) have particle sizes above 1000 μm .
 - 63. The process as claimed in claim 62 wherein less than 5% by weight of the solids have the particle sizes.
- 64. The process as claimed in claim 62 wherein less than 1% by weight of the solids have the particles sizes.
- 25 65. The process as claimed in any of claims 1 to 64, wherein less than 15% by weight of the solids used in the deformable mass(es) have particle sizes above 800 μm .
- 30 66. The process as claimed in claim 65, wherein the amount is less than 10% by weight.
 - 67. The process as claimed in claim 65, wherein the amount is less than 5% by weight.
 - 68. The process as claimed in any of claims 1 to 67, wherein the water content of the tablets is from

50 to 100% of the calculated water binding capacity.

- 69. The process as claimed in any of claims 1 to 68, wherein the deformable mass(es) in the course of processing has (have) a water content of from 2.5 to 30% by weight, based on the mass.
- 70. The process as claimed in claim 69, wherein the water content is from 5 to 25% by weight.
 - 71. The process as claimed in claim 69, wherein the water content is from 7.5 to 20% by weight.
- 15 72. The process as claimed in any of claims 1 to 71, wherein the deformable mass(es) is (are) hardened by means of time-delayed water binding.
- 73. The process as claimed in any of claims 1 to 72, wherein the deformable mass(es) is (are) hardened by means of cooling below the melting point.
- 74. The process as claimed in any of claims 1 to 73, wherein the deformable mass(es) is (are) hardened by means of evaporation of solvents.

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- 75. The process as claimed in any of claims 1 to 74, wherein the deformable mass(es) is (are) hardened by means of crystallization.
- 76. The process as claimed in any of claims 1 to 75, wherein the deformable mass(es) is (are) hardened by means of chemical reaction(s), especially addition polymerization.
- 77. The process as claimed in claim 76, wherein the chemical reaction is addition polymerization.

- 78. The process as claimed in any of claims 1 to 77, wherein the deformable mass(es) is (are) hardened by means of a change in the rheological properties.
- 79. The process as claimed in any of claims 1 to 78, wherein the deformable mass(es) has (have) total surfactant content of less than 5% by weight, based on the mass.

- 80. The process as claimed in claim 79, wherein the surfactant content is less than 4% by weight.
- 81. The process as claimed in claim 79, wherein the surfactant content is less than 3% by weight.
 - 82. The process as claimed in claim 79, wherein the surfactant content is less than 2% by weight.
- 20 83. The process as claimed in any of claims 1 to 82, wherein at least one of the deformable masses comprises bleaches from the group of the oxygen or halogen bleaches, in amounts of from 2 to 25% by weight, based on the mass.
- 84. The process as claimed in claim 83, wherein the bleach is a chlorine bleach.
- 85. The process as claimed in claim 83, wherein the bleach is sodium perborate or sodium percarbonate.
 - 86. The process as claimed in any of claims 83 to 85, wherein the amounts are from 5 to 20% by weight.
- 35 87. The process as claimed in any of claims 83 to 85, wherein the amounts are from 10 to 5% by weight.

The process as claimed in any of claims 1 to 87, 88. wherein at least one of the deformable masses comprises bleach activators from the groups of polyacylated alkylenediamines, especially tetra-(TAED), N-acyl imides, acetylethylenediamine especially N-nonanoylsuccinimide (NOSI), acylated n-nonanoylespecially phenolsulfonates, (n- or iso-NOBS) isononanoyloxybenzenesulfonate N-methylmorpholiniumacetonitrile and sulfate (MMA), in amounts of from 0.25 to 15% by 10 weight, based on the mass.

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- The process as claimed in claim 88, wherein the 89. amounts are from 0.5 to 10% by weight.
- The process as claimed in claim 88, wherein the 90. amounts are from 1 to 5% by weight.
- The process as claimed in any of claims 1 to 90, 91. wherein at least one of the deformable masses 20 comprises silver protectants from the group of the bisbenzotriazoles, benzotriazoles, triazoles, alkylaminotriazoles and the aminotriazoles, transition metal salts or transition metals in amounts of from 0.01 to 5% by complexes, 25 weight, based on the mass.
- The process as claimed in claim 91, wherein the 92. and/or benzotriazole protectant is a alkylaminotriazole complex. 30
 - The process as claimed in claims 91 or 92, wherein 93. the amounts are from 0.05 to 4% by weight.
- The process as claimed in claims 91 or 92, wherein 94. 35 the amounts are from 0.5 to 3% by weight.

- 95. The process as claimed in any of claims 1 to 94, wherein at least one of the deformable masses further comprises one or more substances from the groups of enzymes, corrosion inhibitors, scale inhibitors, cobuilders, dyes and/or fragrances in total amounts of from 6 to 30% by weight, based on the mass.
- 96. The process as claimed in claim 95, wherein the total amounts are from 7.5 to 25% by weight.

- 97. The process as claimed in claim 95, wherein the total amounts are from 10 to 20% by weight.
- 15 98. The process as claimed in any of claims 11 to 97, wherein one of the deformable masses comprises bleaches while another deformable mass comprises bleach activators.
- 20 99. The process as claimed in any of claims 11 to 98, wherein one of the deformable masses comprises bleaches while another deformable mass comprises enzymes.
- 25 100. The process as claimed in any of claims 11 to 99, wherein one of the deformable masses comprises bleaches while another deformable mass comprises corrosion inhibitors.
- 30 101. The process as claimed in any of claims 11 to 100, wherein one of the material strands emerging from the emergence apertures comprises enzymes.
- 102. The process as claimed in claim 101, wherein the enzyme-containing material strand is enveloped by an enzyme-free material.

- 103. The process as claimed in any of claims 11 to 102, wherein one of the deformable masses comprises bleaches while another deformable mass comprises surfactants.
- 104. The process as claimed in claim 103, wherein nonionic surfactants are present.

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- 105. The process as claimed in claim 103, wherein alkoxylated alcohols having 10 to 24 carbon atoms and from 1 to 5 alkylene oxide units are present.
- 106. The process as claimed in any of claims 11 to 105, wherein at least two deformable masses comprise the same active substance in different amounts.
 - 107. The process as claimed in any of claims 1 to 106, wherein the deformable mass(es) comprise(s) a paraffin wax having a melting range of from 50°C to 55°C.
- 108. The process as claimed in any of claims 1 to 107, wherein the plastically deformable mass(es) comprise(s) at least one substance from the group of polyethylene glycols (PEG) and/or polypropylene glycols (PPG).
- 109. The process as claimed in any of claims 1 to 108, wherein the tablets comprise less than 10% by weight of free water.
 - 110. The process as claimed in claim 109, wherein the free water is less than 5% by weight.
- 35 111. The process as claimed in claim 109, wherein the free water is less than 1% by weight.

- 112. The process as claimed in claim 109, wherein the free water is less than 0.5% by weight.
- 113. The process as claimed in any of claims 1 to 112, wherein the tablets have a density of more than 800 kgdm⁻³.
 - 114. The process as claimed in claim 113, wherein density is more than 900 kgdm⁻³.
- 115. The process as claimed in claim 113, wherein density is more than 1000 kgdm⁻³.

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- 116. The process as claimed in claim 113, wherein density is more than 1000 kgdm⁻³.
 - 117. The process as claimed in any of claims1 to 116, wherein the tablets are subjected to an aftertreatment step.
- 118. The process as claimed in claim 117, wherein the aftertreatment step comprises the coating of the tablets with a pourable material.
- 25 119. The process as claimed in claim 118, wherein the pourable material has a viscosity <5000 mPas.
- 120. The process as claimed in either of claims 117 to 119, wherein the aftertreatment step comprises an additional shaping step.
 - 121. The process as claimed in claim 120 wherein the shaping step is impression.
- 35 122. A laundry detergent or cleaning product tablet comprising at least 30% by weight of phosphate(s), wherein the water content of the tablet is from 50 to 100% of the calculated water binding capacity.

- 123. The tablet as claimed in claim 122, which comprises at least 40% by weight, based on the tablet weight.
- 124. The tablet as claimed in claim 123, wherein at least 45% by weight of phosphate is present.

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- 125. The tablet as claimed in claim 123, wherein at least 50% by weight of phosphate is present.
 - 126. The tablet as claimed in any of claims 122 to 125, which comprises alkali metal phosphate(s) in amounts of from 30 to 80% by weight, based on the tablet weight.
 - 127. The tablet as claimed in claim 126, wherein the alkali metal phosphate is pentasodium and/or pentapotassium triphosphate.
- 128. The tablet as claimed in claim 126, wherein the alkali metal phosphate is sodium and/or potassium tripolyphosphate.
- 25 129. The tablet as claimed in claims 126 to 128, wherein the amounts are from 35 to 75% by weight.
 - 130. The tablet as claimed in claims 126 to 128, wherein the amounts are from 50 to 70% by weight.
 - 131. The tablet as claimed in any of claims 122 to 130, wherein the water content of the tablet is from 55 to 95% of the calculated water binding capacity.
- 35 132. The tablet as claimed in claim 131, wherein the water content is from 60 to 90%.

- 133. The tablet as claimed in claim 131, wherein the water content is from 65 to 85%.
- 134. A laundry detergent or cleaning product tablet
 whenever prepared by the process as claimed in one
 or more of claims 1 to 121.

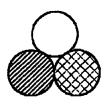


Fig. 1

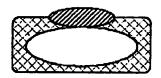


Fig. 2

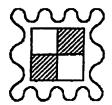


Fig. 3

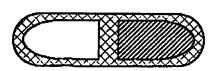


Fig. 4



Fig. 5

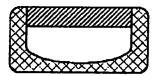


Fig. 6



Fig. 7

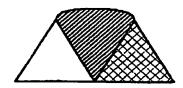


Fig. 8

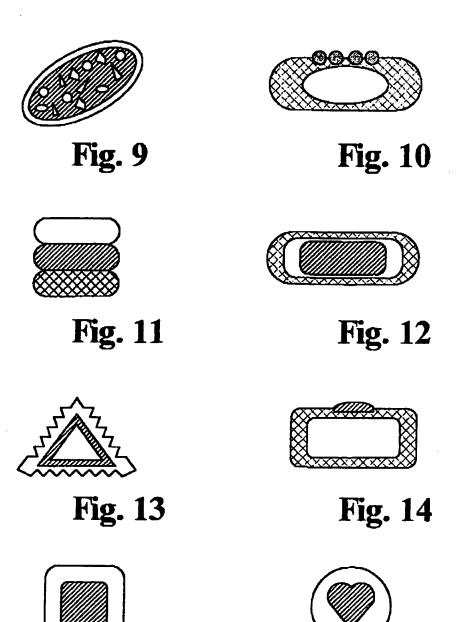


Fig. 16

Fig. 15

